# Anti-multipactor TiN coating of RF power coupler components for TESLA at DESY

(Description of a temporary coating setup, processing parameters and TiN coating impact on RF performance of couplers)

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## Abstract

A development project was carried out at DESY, aimed at reducing secondary electron emission and multipactor effects in TESLA couplers by TiN layers generation on surfaces which were not protected in this way previously. Thin TiN films on ceramic or metallic surfaces were produced by deposition from Ti vapor in low pressure ammonia. Selection of processing parameters and their effect on multipactor suppression in RF field have been studied using a multipactor test resonator at DESY. Appropriate values of deposition rate, substrate temperature, final layer tickness and chemical conversion procedure were selected. A significant reduction of multipactor time during RF tests was reached due to surface coating. Chemical analysis of TiN layers on both ceramic and metallic substrates has been performed using SIMS method. TiN coating of more than 80 waveguide and cylindrical RF windows were done for TTF2, TTF3 and TTF4 versions of TESLA couplers. Surface processing of flat waveguide windows for TTF2 resulted in significant reduction of multipactor effects and improvement of power transmission. Their good performance remained unchanged after 24-hour exposition to air. TiN coating of all vacuum-facing surfaces of a TTF2 coupler led to reduction of the indispensable RF conditioning time at a pulse length of 20 μs from typically 3 days down to 4.5 hours. The above activities are considered a preparatory step for the future large-scale anti-multipactor protective coating of TESLA couplers.

# 1 Introduction

The performance of RF power elements is often limited by field emission and multipacting. The latter is initiated if certain resonance conditions are fulfilled for trajectories of electrons striking surfaces of bigger than 1 secondary electron yield. Substantial multipactor currents absorb a large part of RF power. The power can be deposited in localized areas which results in inhomogeneous heating, thermal stresses and, particularly in ceramic windows, in cracking.

Along with careful design of coupler and window geometries which is aimed at avoiding resonance conditions, secondary electron emission coefficient of vacuum-facing surfaces is reduced by applying thin layers of special materials. A considerable effort has been done to study secondary emission, anti-multipactor and chemical properties of such compounds as chromium oxide, titanium nitride and titanium oxinitrides ([1] – [10]). Currently TiN thin layers are the most commonly used for multipactor suppression due to their low secondary emission on the one hand and good stability in RF field on the other.

Apart from various modifications of sputtering methods, evaporation technique is used for TiN layers generation. Good results were reached using titanium vapor deposition in a reactive atmosphere of low pressure ammonia. The vapor is usually released by sublimation from electrically heated titanium filament. Using the latter method multipactor phenomena were successfully eliminated from RFQ accelerator at Los Alamos [11] and coupler windows at FERMILAB [12].

In response to multipactor problems connected with the construction of TESLA couplers at DESY, sublimation technique was implemented and developed in the years 1999-2003 using a temporary coating device. Along with TiN coating of several types of ceramic coupler windows, TiN deposition on all the vacuum-exposed TTF2 coupler surfaces has been done.

# 2 Processing device and method

## 2.1 Apparatus and sublimation setup

A temporary TiN coating setup consists of a cylindrical stainless vessel of 20 cm diameter and about 80 cm tall, equipped with ports for power supply and diagnostic feedthroughs, quartz windows and gas inlet valves. It is connected to a pumping system based on a turbopump of 500 l/s pumping speed.

The sublimation sources (filaments) are used, made of titanium wires (99.8% purity), 1 mm in diameter. They are energized from three 30V/30A dc power supplies via 4.5mm diam. copper rods and feedthroughs. Each RF power element to be coated requires a specific filaments geometry, screens and deposition sequence. In order to desorb impurities a fresh filament requires preheating up to ca. $1000^{o}$ C, after which it losses its flexibility due to the titanium phase transition at $882.5^{o}$ C. At a typical sublimation temperature (ca. $1500^{0}$ C) in turn, the filament is no more rigid, elongates itself and often changes its position due to thermal stresses and high temperature creep under its own weight. The filaments are best used when hanging as horizontal loops (catenaries) or vertical lines. In order to compensate for thermal stresses and displacement of a catenary the electrical connections are often made of stainless steel contacts attached through flexible elements (vertically suspended flexible copper strings or bands) to the setup structure via steatite or aluminum insulators. Horizontal titanium wire loops are fixed in several points by titanium wire supports connected to stainless screws attached to ceramic insulators. These supports withstand sublimation temperatures and do not bring about a substantial heat drain.

Vertical lines in turn are stretched between two massive, electrically insulated stainless current contacts, the upper one being equipped with a spring mechanism in order to compensate for wire elongation and thus avoid lateral displacement of the wire. An additional dc power supply was often used to establish a constant voltage between the filament and the rest of the vacuum chamber and metallic parts of the coated RF elements. An optical pyrometer is used to determine the temperature of the filament. The pyrometer is based on the single 650 nm wavelength , which requires surface emissivity knowledge (ca. 0.46 for titanium at typical sublimation temperatures) to apply appropriate corrections. It sees the filament through a 4 mm thick quartz window of a 0.93 transmission factor.

Two gas-handling tubing systems are used, both connected via pressure reducers to the cylinders with 99.999% purity nitrogen and 99.98% purity ammonia, respectively. The nitrogen is used for purging and venting the system and the ammonia -for chemical conversion of the layer. Two variable leak valves are used for precise nitrogen and ammonia admission into the vessel and one more valve - for fast venting. To assure continuous gas pressure measurement from atmospheric down to the basic vacuum there are two Penning pressure gauges and a Pirani-type gauge. Measurement of residual gas composition was performed using a quadrupole mass spectrometer QUADRUVAC PGA 100.

Layer thickness distribution is calculable using basic emission laws. Some of the calculation results obtained for our coating system are presented in chapter 4. Due to space restrictions we had largely to give up using a film thickness monitor based on the resonant frequency of quartz crystals. In case of the source-crystal distance lower than 25 cm the crystal frequency vs. deposited layer thickness dependence is strongly disturbed by heat and radiation [13]. To have a guiding parameter during the exposure we performed an on-line measurement of the resistance of a TiN layer deposited on a separate

ceramic plate installed in the setup at a proper distance from the filament. The apparatus was equipped with a NiCr-Ni thermo element and electric thermometers to monitor the temperature of a substrate and its environment during and after deposition. The substrate temperature influences the nature of deposition and effects the Ti $\rightarrow$ TiN chemical transition within the layer.

## 2.2 Exposure calculations

### 2.2.1 Sublimation rate

The temperature of a filament varies along its length due to its contact with current leads and supports. The considerations below refer to the filament temperature far away from the points where supports and contacts cause heat drain. We also assume that sublimation time is short enough to avoid any significant reduction of the cross section of the wire , which might change its resistance and the heating power supplied. We have
evidence that even partial Ti $\rightarrow$ TiN conversion on the filament surface in ammonia reduces the sublimation rate. Therefore, as a rule, we changed filaments after each two or three coating operations (or latest after a total sublimation time of 5 min).

Filament temperature dependence on heating current was measured directly by using the optical pyrometer. The measurements were performed with a 30 cm long vertical titanium wire of 1 mm diam. at typical working parameters, including ammonia flow at a pressure of $10^{-3}$ mbar. The results are shown in Figure1a. A least-square fit to the measured data gave the linear expression between absolute temperature of the wire $T_w$ (in K) and the filament dc current I (in A):

$$T_w = 58.9\, I + 825 \qquad (1)$$

The formula has been derived for typical current values falling between 13 and 17 A. The working temperature of the filament has been chosen as a compromise between a required deposition rate and the wire lifetime.

The titanium vapor pressure p (torr) and the rate of sublimation are given by the following formulas [14]:

$$\mathrm{Log}\, p = (0.009202 T_w - 19.72) \qquad (2)$$

$$w = 0.585\, p(M_{Me}/T_w)^{1/2} \qquad (3)$$

where $M_{Me}$ is the atomic weight of the metal (47.9 for Ti) and w (in kg/($m^2$s)) – the titanium mass sublimated per unit wire surface area, per unit time. $T_w$ has the same meaning as in (1). The resulting sublimation rate vs. current relationship, obtained from (1) – (3), is given as a continuous line in Figure 1b. The experimental values of average sublimation rates derived from the mass decrease of vertical filaments after 10 min

operation are given as open circles in the plot. The computed and measured values do not differ by more than 20%.

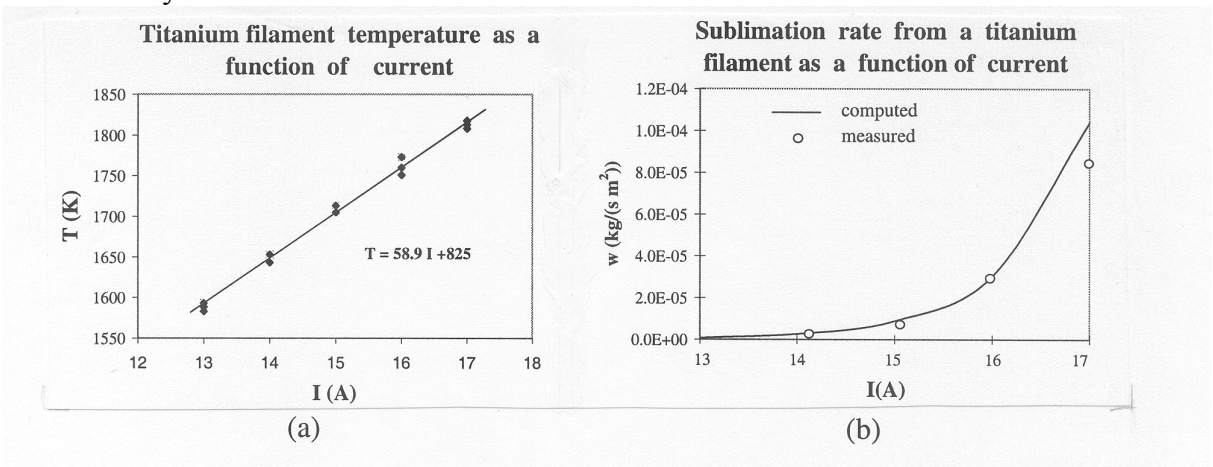



Figure 1: Dependence of (a) 1 mm diam. Titanium filament temperature and (b) corresponding sublimation rate on heating current.

### 2.2.2 Calculation of the layer thickness distribution

The geometrical part of exposure computations, leading to deposit thickness distribution, has been done using emission laws. To assess the resulting layer uniformity, a linear or ring-shaped wire source can be represented by an array of point sources [15]. In our calculations a sufficiently short filament section (typically 2-3 mm long) was represented by an idealized point source of adequate emission rate. The amount of deposit $(dM_s/dA_s)_i$, received by a substrate surface element $dA_s$ from the i-th wire section $dW_i$ (Fig. 2) is thus approximated by:

$$(dM_s/dA_s)_i \approx (M_e \cdot \cos\Theta_i)/(4\pi \cdot r_i^2)$$

where $M_e$ is the total mass of material evaporated from a single section, whereas $\Theta_i$ and $r_i$ are the angle of incidence and the distance between element $dA_s$ and the i-th filament section, respectively.

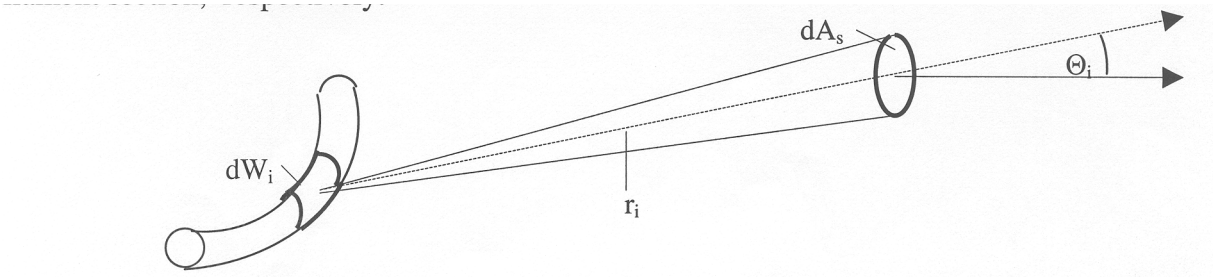



Figure 2: Evaporation from a wire section $dW_i$ onto a substrate surface element $dA_s$.

The total layer thickness d on $dA_s$, reached after deposition time t is given by:

$$d \approx w \cdot s_e \cdot t \, (\Sigma(\cos \Theta_i / r_i^2)/(4\pi\rho_{Me})) \qquad (4)$$

where w is the sublimation rate given by (3), $s_e$ – emission surface of a single filament section and $\rho_{Me}$ denotes density of metal film. The summation extends over all the wire sections. In practice the computation was performed using EXCEL Worksheet capabilities.

The „reversed square law ($\sim 1/r^2$)" for deposition from a point source corresponds to „$\sim 1/r$" dependence of deposition rate on the distance r from a sufficiently long straight filament. This „1/r" law has been positively verified by us only after biasing the filament at + 10 V respectively the (grounded) substrate. The plots in Figure 3 show the theoretical deposition rate versus distance relationship for a 30 cm long, vertical titanium wire at a current of 16 A (continuous line). The points refer to titanium deposition rates on glass samples located at different positions respectively the filament. These values were obtained from the apparent „weight increment" of the coated samples after 1-hour deposition. The change of deposition rate over this time was checked using a quartz crystal thickness monitor and taken into account. The tests were performed with normal ammonia flow through the system without (Fig. 3a) and with (Fig. 3b) positive bias of the filament. A better compliance with „1/r" law in case of the biased wire is clearly visible. The positive impact of the bias is also reported in [12]. The nature of this effect is not fully understood yet.

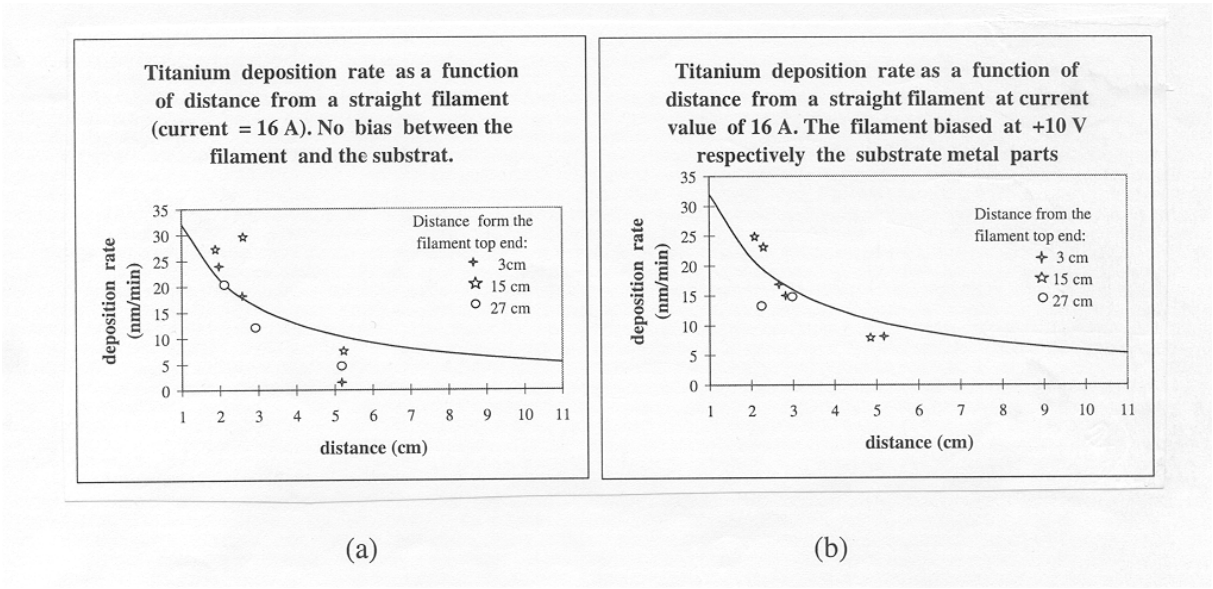



Figure 3: Titanium deposition rate dependence on the distance from a vertical, 30 cm-long filament (a) without nad (b) with 10 V positive bias of the filament.

### 2.2.3 Ti deposition conditions

The number of metal particles colliding with a unit substrate surface per unit time is given by [16]:

$$dN_{Me}/(A_s dt) = N_A \cdot \rho_{Me} \cdot c_{Me}/M_{Me} \quad (5)$$

were $dN_{Me}/(A_s dt)$ (atoms/(cm$^2$ s)) is the number of metal atoms impinging a substrate surface of $A_s$ area per unit time, $N_A$ (= $6.024 \cdot 10^{23}$) – Avogadro's number, $\rho_{Me}$ – density of metal film ($\leq$ 4.54 g/cm$^3$ for Ti), $c_{Me}$(cm/s) – pure metal condensation rate and $M_{Me}$(g/mol) – molar mass of the metal (= 47.9 g/mol for Ti).

According to the „1/r" relationship, at the biggest source-substrate distance of 5 cm and filament current equal 16 A, the lowest practically reached titanium layer growth rate was close to $0.74 \cdot 10^{-8}$ cm/s (4.5 nm/min). The corresponding collision rate of Ti atoms is ca. $4.2 \cdot 10^{14}$ atoms/(cm$^2$ s).

In the deposition of titanium films the background pressure is kept as low as possible. The interaction of residual gases, especially oxygen and water vapor, with strongly gettering titanium has detrimental effects on film properties. Oxygen is chemisorbed inside the layer and oxides are formed. Titanium oxides content in surface layer should be reduced. Their thermal stability is reported to be lower than that of TiN ([1] and [17]). Partial pressures of the main residual gas constituents and ammonia are given in Table 1. The residual gas mass spectrum was measured with the mass spectrometer at a basic pressure (before starting the coating procedure) of $10^{-6}$ mbar. The data for ammonia and titanium vapor relate to typical coating conditions.

Table 1: Typical partial pressures and impingement rates of ammonia, titanium and the main residual gas constituents in the titanium sublimation setup.

| | Residual gas (total pressure $10^{-6}$ mbar) | | | | Coating conditions | |
|---|---|---|---|---|---|---|
| Molecular mass | 2 ($H_2$) | 18 ($H_2O$) | 28 ($N_2$) | 32 ($O_2$) | 17 ($NH_3$) | 48 (Ti) |
| Partial pressure (mbar) | $8.5 \cdot 10^{-7}$ | $8 \cdot 10^{-8}$ | $4 \cdot 10^{-8}$ | $7.2 \cdot 10^{-9}$ | $1 \cdot 10^{-3}$ | |
| Impingement rate (cm$^{-2}$ s$^{-1}$) at 350 K | $8.6 \cdot 10^{14}$ | $2.7 \cdot 10^{13}$ | $1 \cdot 10^{13}$ | $1.8 \cdot 10^{12}$ | $3.5 \cdot 10^{17}$ | $4.2 \cdot 10^{14}$ |

For pressures given in mbar the collision rate of gas molecules with substrate surface is given by [16]:

$$dN_g/(A_s dt) = 2.67 \cdot 10^{22} \cdot (M_g \cdot T_g)^{-1/2} p_g \quad (6)$$

were $dN_g/(A_s dt)$ (molecules/(cm$^2$ s)) denotes the number of gas molecules colliding with a unit substrate area per unit time and $M_g$ (g/mol), $T_g$ (K), $p_g$ (mbar) are molecular mass, absolute temperature and pressure of gas, respectively. The impingement rate for oxygen at a pressure of $7.2 \cdot 10^{-9}$ mbar (Table 1) and a temperature of 350 K is smaller than $2 \cdot 10^{12}$ cm$^{-2}$ s$^{-1}$. Taking the above estimate for Ti atoms collision rate with a substrate $O_2$ : Ti impingement ratio is well below 1 : 200. The titanium condensation coefficient equals 1 and is somewhat higher that of oxygen [16]. It

follows then that the amount of oxygen incorporated from residual gas within the titanium layer is safely low. Water vapor is much more abundant – its impinging rate amounts to 7% of that of titanium (Table 1). Ceramic substrates, however, are typically coated after reaching a temperature of ca. $150^{o}$ C which assures the on-line water baking.

The coated components are exposed to the atmosphere after processing, which leads to surface oxidation and water adsorption. The resulting growth of secondary electron yield of the surface is mainly determined by the presence of surface water and hydrocarbons [9].

## 2.3 Coating operation

All the parts internal to vacuum are cleaned in an ultrasonic bath, first in a solution of a degreasing agent (soap) , next in ultra-pure water and finally dried with pure nitrogen blow. The assembly of the setup is performed so as to avoid fingerprints or impurities collection on surfaces. Sometimes ceramic surfaces require preliminary sandblasting.

Vacuum pumping is normally preceded by three times evacuating down to $10^{-1}$mbar and refilling with pure nitrogen. A basic pressure of $2 \cdot 10^{-6}$ mbar is typically reached after 4–5 hours of pumping. Then the filaments are preheated for 0.5-3 hours by dc current flow to a temperature of ca.$900^{0}$ C to desorb impurities. The vessel is filled with ammonia up to about $10^{-1}$mbar and evacuated again. The time dependence of the ammonia pressure, filament current and substrate temperature are shown in Figure 4. After the basic vacuum is reached once more the wire temperature is rised to roughly 1000 - $1100^{0}$ C and maintained for 10 - 20 min. to increase the substrate temperature up to 130-$170^{0}$ C. The substrate preheating was not performed in case of possible thermal stresses within a coated element (e.g. waveguide windows) or when coating metal components. At $1100^{0}$ C titanium deposition is still negligible during a sufficiently short time. Just before coating the ammonia pressure is rised (under continuous pumping) to a value corresponding to a mean free path of titanium atoms close to average filament- substrate distance (FSD). The optimum ammonia pressure $p_a$ is given by the formula [18]:

$$FSD = 3 \cdot 10^{-20} \cdot T_g(K)/(2.54 \cdot p_a(mbar) \cdot (4.42 \cdot 10^{-8})^2) \qquad (7)$$

The pressure corresponding to a typical filament- source distance of 2 – 6 cm is within $10^{-4}$ – $10^{-3}$ mbar region. The ammonia is admitted through a precision leak valve while the vacuum vessel is pumped.

The coating is started by rising the heating current up to 14 – 16 A for a time determined by exposure calculation (typically 0.5 – 3 min). The filament temperature is measured with the pyrometer. If several, separately fed filaments are used, they are heated in sequence. As a check, surface layer resistance can be on-line measured on a reference ceramic sample supplied with two parallel conducting lines on

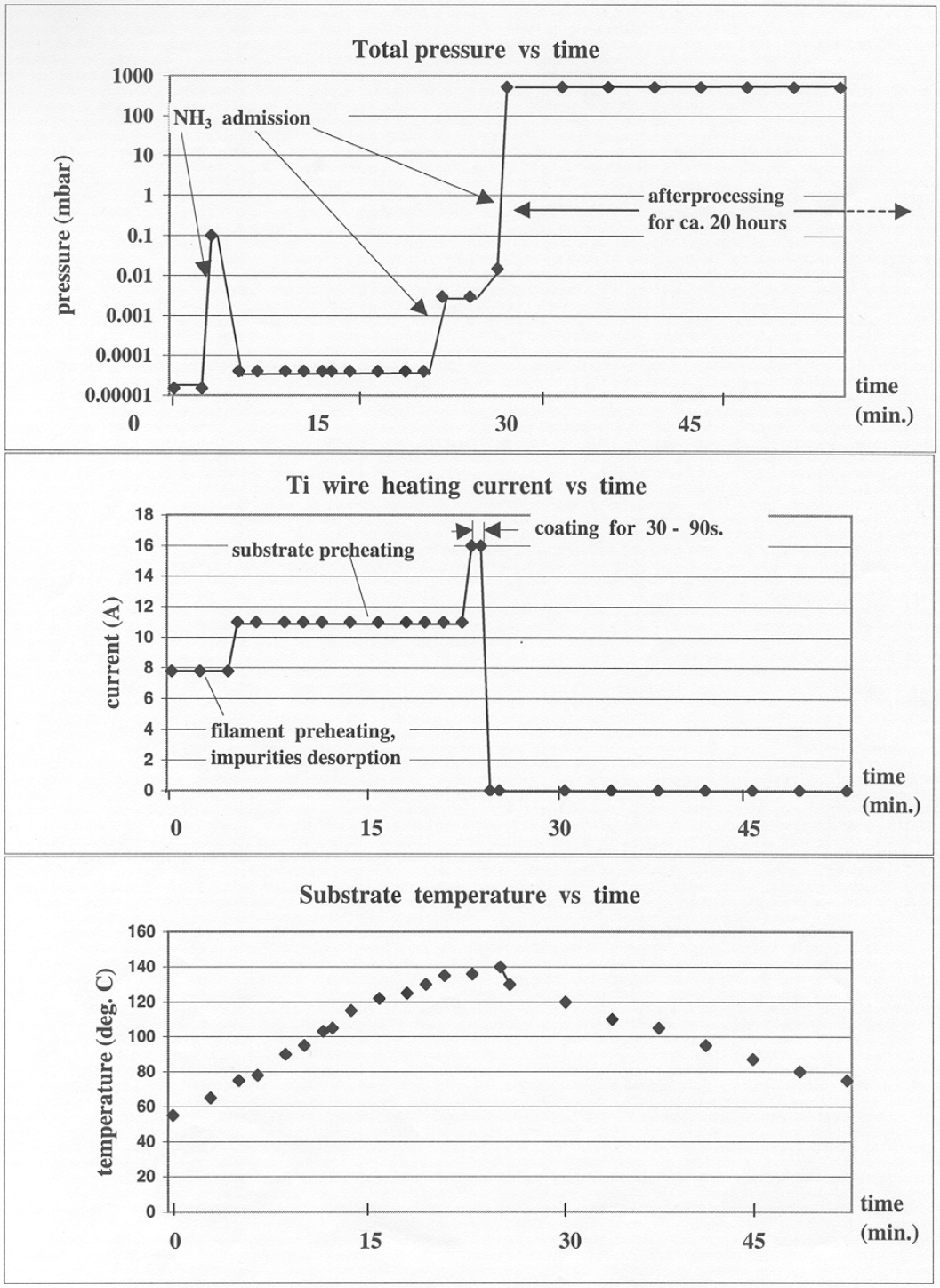



Figure 4: Time schedule of cylindrical windows TiN coating for TTF3 coupler.

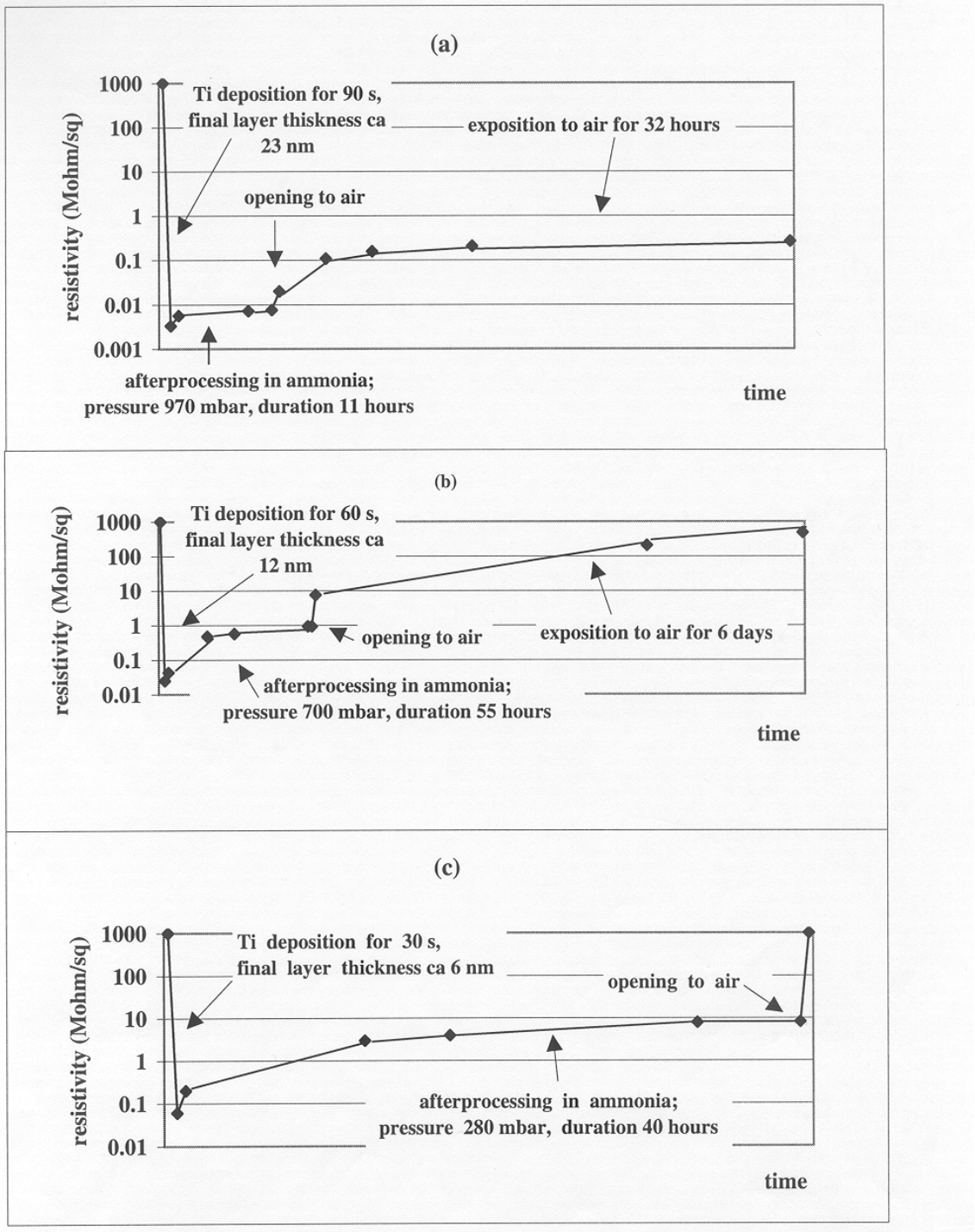



Figure 5: Surface layer resistivity as a guide parameter for titanium deposition and further chemical processing of the layer. Titanium deposition has been interrupted at the following minimum resistivity values: (a) 3.7 kOhm/sq, (b) 25 kOhm/sq and (c) 60 kOhm/sq. The time scale of the plots was shortened so as to mach the processing time to the chart length.

the exposed surface and a pair of contacts. The surface layer resistivity was used as an additional guide parameter in order to terminate the coating process at a layer thickness sufficiently small to enable further Ti → TiN conversion in ammonia atmosphere. Deposition started typically at a resistivity of some GOhm/sq and was interrupted at 10-100 kOhm/sq. Further drop of the layer resistance brings the risk of incomplete chemical conversion, high residual layer conductance (even after exposition to air) and hence – excessive ohmic losses in RF field. The resistivity measurement, however, had only a relative value for absolute layer thickness determination. The layer conductivity depends on substrate temperature and surface interaction with gas. The thickness distibution and deposition rate were checked during preliminary coating tests, when the RF component to be coated was substituted by an array of strategically exposed alumina samples. The layer thickness after titanium deposition extended to several tenths of minutes was derived from the apparent „mass growth" of each sample determined by weighing.

After deposition pumping of the vessel is stopped and the ammonia pressure is increased to 150 – 900 mbar to enhance the chemical conversion. The conversion is monitored via the layer resistivity growth. A rapid resistance growth, which takes place within few hours after coating is next, followed by slow increase, approximately linear with time. The rate of this increase of layer resistivity depends strongly on its minimum value at the end of deposition. Figure 5 shows variation with time of layer resistivity for different coating trimes. The three plots refer to minimum resistivity values of 3.7, 25, and 60 kOhm/sq. In the first case (Fig. 5a) the layer resistance increases very slowly in time during final processing in ammonia and saturates below 10 kOhm/sq. After exposition to air it grows further and saturates at 200 kOhm/sq within some hours. That high conductivity of the layer cannot be accepted. Completion of titanium deposition at 25 or 60 kOhm/sq (Fig.5 b,c) resulted in resistivity growth in ammonia to 1 and 10 MOhm/sq, respectively. Further exposition of these two samples to air caused rapid oxidation of residual titanium in the surface layer and their surface conductivity dropped to low values (of the order of $(GOhm/sq)^{-1}$) within 1 day.

Processing in ammonia is normally followed by pumping the vessel down to $10^{-2}$ mbar followed by filling it with pure nitrogen and opening to air. The coated RF elements are then collected in a cryostat or in a vessel with nitrogen.

## 3 RF behaviour of TiN coated samples - preliminary studies

The influence of TiN coating on multipactor suppression in RF field was studied using a test resonator at DESY [20]. The device enables a straightforward measurement of RF multipacting current between two flat electrodes installed in a specially designed coaxial resonator. The resonance condition for two-surface, first order multipacting is reached at a frequency of 500 MHz. The cavity is operated at RF power of 5 W above the onset of

multipacting. During the test the multipactor current is reduced and the RF electric field in the cavity increases until the electron current disappears completely. For each measurement a new pair of electrodes is installed after surface processing. The time needed to overcome multipacting is taken as a figure of merit of the applied coating procedure.

Ten pairs of copper or aluminum electrodes were Ti -coated from vertical titanium filaments in ammonia in accordance with the procedure described in 2.3. In addition ceramic samples were coated in parallel with the electrodes. The resistivity of the surface layers deposited on these samples was on-line measured. The ceramic samples and electrodes occupied equivalent positions respectively the filaments. The deposition rate has been varied between 8 and 20 nm/min and the final layer thickness - between 4 and 20 nm. The substrate temperature was 25 or 1500 C. The latter was reached by resistive heating of the vacuum vessel. For chemical conversion of titanium, further processing in high pressure ammonia (150 - 900 mbar) was applied.

RF tests enabled selection of the optimal coating conditions and final thickness of the layer. RF performance of the layers of thickness between 7 and 15 nm, reached in a single coating operation, proved to be most promising. The corresponding multipactor times were suppressed to 5 - 12 min whereas for a pure (non-coated) copper surface it was close to 50 min and for pure aluminum it's practically unlimited.

A 4 nm thick layer was too thin to suppress secondary electron emission effectively (multipactor time of 50 min). On the other hand a very thick layer of ca. 40 nm, deposited in ammonia, brought a sharp drop of the multipactor time (down to 30 s) but exhibited an excessive residual conductivity ((4 kOhm/sq)-1) due to low degree of Ti$\rightarrow$TiN conversion. We did not succeed in enhancing titanium conversion to nitride by subdividing the deposition into several steps, each followed by processing in ammonia. Also rising the substrate temperature to $150^0$ C by heating the vessel did not improve RF performance. One of the electrodes processed in the heated vessel showed a multipactor time of more than 2000 s. The latter effect might be attributed to enhanced desorption of impurities from the hot vessel walls. Heating the vessel was accompanied by a rise of residual gas pressure.

Most ceramic samples coated together with RF electrodes showed yellowish or grey-yellowish color after exposition to air, indicating partial Ti $\rightarrow$TiN transition. The resistivity of layers grew up from typically 10 - 60 kOhm/sq to several hundreds kOhm/sq as a result of final processing in ammonia. After exposing to air it grew further to tenths or hundreds of MOhm/sq over a few days, depending on layer thickness. In most cases cooling the coated ceramic samples down to 70 K in liquid nitrogen reduced further their surface resistivity by a factor of 10 -60. This effect is likely to reduce cryogenic losses in cold (70 K) coupler windows. The negative values of resistance temperature coefficient are typically attributed to incorporation in the lattice of impurity phases like nitrides or oxides [21]. Selected, representative results of the tests are given in Table 2.

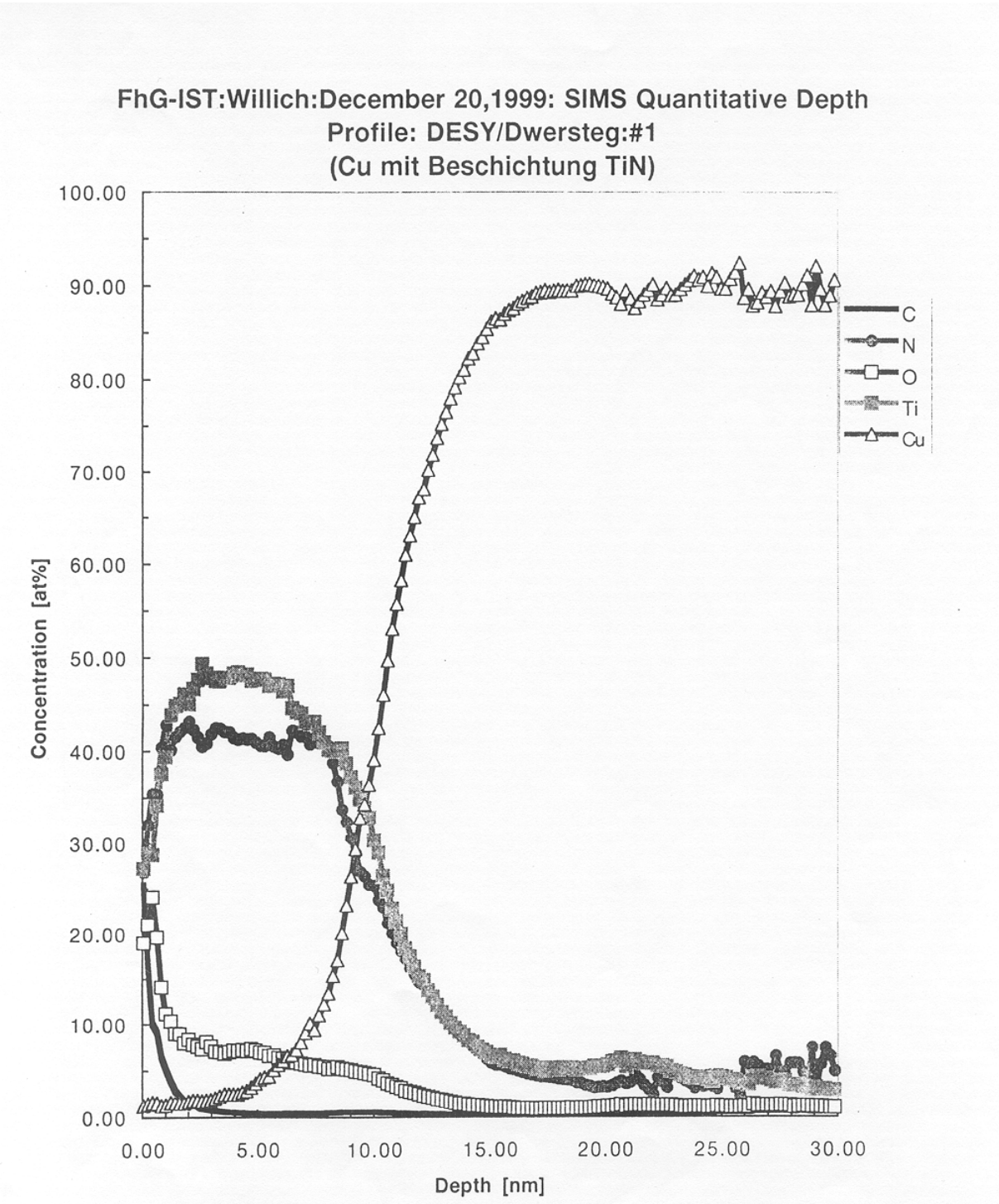



Figure 6: SIMS quantitative depth profile for 8 nm thick TiN layer on copper according to [19].

Table 2: Test results from DESY coaxial test resonator and resistivities of surface layers on ceramic samples.

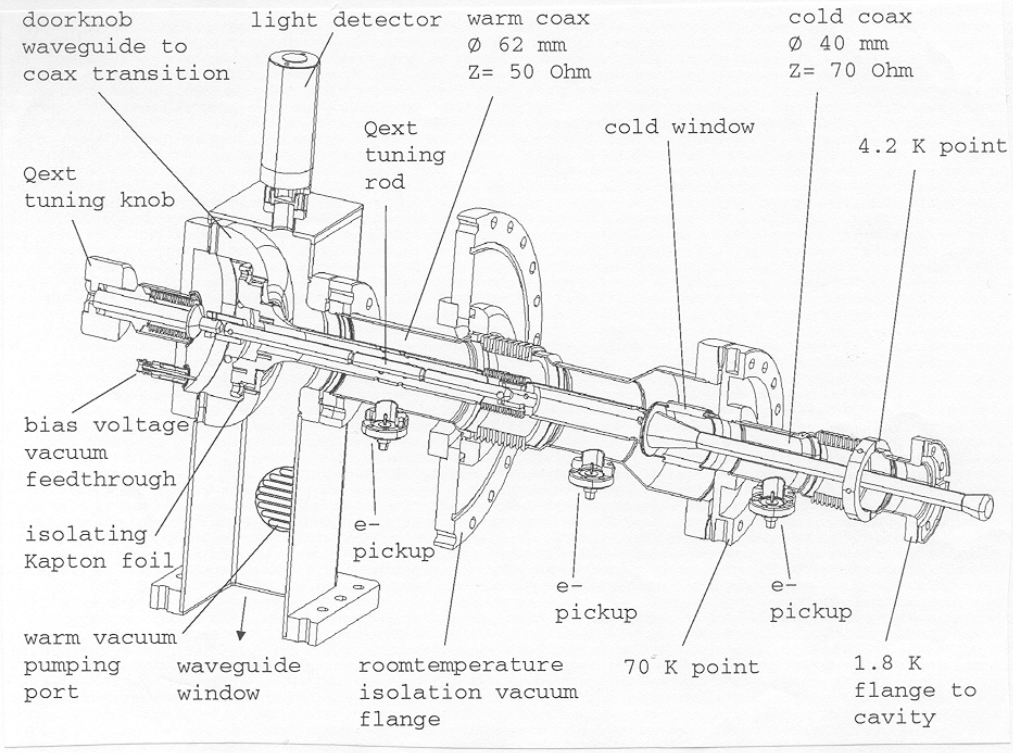
| Substrate material | Vessel temp. | Final layer thickness | After processing: $NH_3$ pressure, time | Multipactor time | Surface layer resistivity on ceramic plates after 4-weeks exposition to air | |
|---|---|---|---|---|---|---|
| | | | | | at 300 K | at 70 K |
| copper | clean surface before coating | | | 3000 s | | |
| aluminum | clean surface before coating | | | >6500 s | | |
| aluminum | $150^0$ C | 15 nm | 300 mbar, 2 days | 800 s | 55 Mom/sq | 1 Gohm/sq |
| aluminum | $150^0$ C | 7 nm | 300 mbar, 2 days | 650 s | 3 Gohm/sq | 200 Gohm/sq |
| aluminum | $25^0$ C | 7 nm | 700 mbar, 1 day | 700s | 24 Gohm/sq | 300 Gohm/sq |
| aluminum | $150^0$ C | 4 nm | 200 mbar, 1 day | 3000 s | | |
| copper | $150^0$ C | 8 nm | 200 mbar, 1 day | 750 s | | |

One of the coated copper electrodes (Table 2) was tested using SIMS method. The received depth profile of an 8 nm-thick surface layer is shown in Fig. 6. The layer elemental composition is dominated by titanium and nitrogen, whereas oxygen contents remains below atomic 10% except for immediate surface region. The N:Ti atomic ratio is pretty close to the stoichiometric (higher than 0.8 : 1).

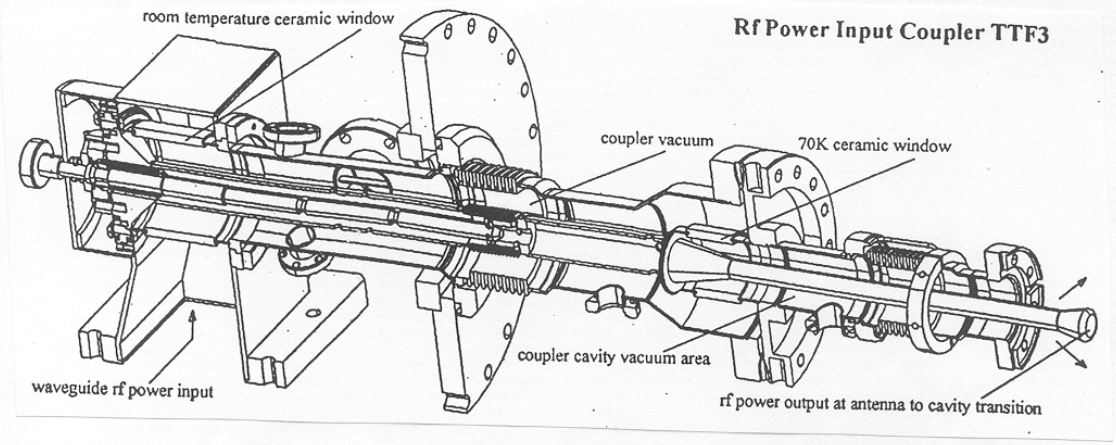
# 4 TiN coating of RF power elements

Tesla Test Facility (TTF) needs RF input couplers , which transfer 1.3 GHz energy from RF distribution system to superconducting cavities. Several different coaxial couplers were manufactured and operated in TTF. TTF2 and TTF3 couplers are mainly used until now. TTF2 coupler design is shown in Figure 7a. An electrically coupling antenna is permanently connected to a cylindrical ceramic cold window as a part of a cold coaxial line of 70 Ohm impedance (40 mm of diameter). $Q_{ext}$ tuning is performed by moving axially the antenna together with the cold window. Their position is regulated through a tuning rod inside the inner conductor of a warm coaxial line (warm coax., 62 mm in diameter, impedance of 50 Ohm). The latter is connected to a doorknob waveguide to coax transition. The doorknob and the inner surface of the end section of the waveguide in TTF2 are exposed to warm vacuum. They are separated from ambient air by a planar waveguide window (not shown in Figure 7a). A large surface exposed to warm vacuum in this coupler rises multipactor risk.

The TTF3 coupler is compatible with TTF2 as it contains similar cold and warm coax design (Figure 7b). Unlike TTF2 it uses a cylindrical warm window in the half height waveguide to coax transition. The waveguide is filled with air in this case and thus the warm vacuum facing inner surface is much reduced. Ten couplers of TTF4 version, with 80 mm diam. coax line, with two cylindrical windows, have been manufactured to be used with superconducting cavities in "superstructure" configuration.




(a)




(b)

Figure 7: Cross-sectional view of (a) TTF2 and (b) TTF3 coupler.

Before operating on a cavity a coupler is preconditioned on a special test-stand at room temperature. After preliminary baking of the system, RF processing is done at traveling wave. The power is varied from low to high levels, beginning with short pulses (20 μs at a repetition rate 1-2 Hz). After achieving a specified power value the pulse length is doubled and the power rise is carried out again. After the final pulse length of 1.3 ms is reached the power is sweeped between zero and 1 MW. Several power levels are localized which correspond to multipactor activity inside the system. Full accounts on test procedure and processing are given in [22] and [23]. The first power rise (at 20 μs pulse length) takes up to 3 days under normal conditions. After RF discharge processing in argon inside the coupler test-stand this time was reduced to 19 hours [22]. The TiN surface anti-multipactor coating was aimed at further reduction of the processing time. It comprised coating of the following components between 1999 and 2003:

* 68 cold (70 K) cylindrical windows for TTF2 and TTF3,
* 66 warm (room temperature) cylindrical windows for TTF3,
* 10 warm and 10 cold cylindrical windows for TTF4,
* 36 planar wave-guide windows for TTF2,
* whole surface coating of a single TTF2 coupler.

## 4.1 TiN coating of cylindrical windows for TTF2,TTF3 and TTF4 couplers

The cold and warm cylindrical windows of alumina for two TTF2, sixty three TTF3 and ten TTF4 couplers were supplied with 1 mm- thick copper collars used for welding with other coupler components. Their cross-sectional views are given in Figure 8. The collars are brazed to metallized areas inside 2 mm-deep grooves machined on both sides of the ceramic rings. The windows were TiN coated successively in two operations:

- coating of the end window surfaces (in the immediate region of copper collars) while protecting the collars from coating (to enable further welding),
- coating of cylindrical surfaces (inner surfaces of warm windows, inner and outer surfaces of 70 K windows).

### (1) Coating of end surfaces

The general scheme of the sublimation setup for end surfaces coating is shown in Figure 9. A window (supplied with stainless steel shielding on ceramic ring and a band of aluminum foil on the collar to protect selected areas) is installed between two horizontal discs (platforms). The window‘s axis is oriented vertically. In order to minimize shadows and layer asymmetry coming from the collars, two titanium wire loops of diameter identical to that of the copper collar, are suspended on insulating supports above the

upper and below the bottom collar of the window at a distance of $h \approx 50$ mm (limited by space restrictions of the vessel) from the substrate surface. In practice the layer thickness achieved on the end surfaces outside the collar is still strongly reduced (by a

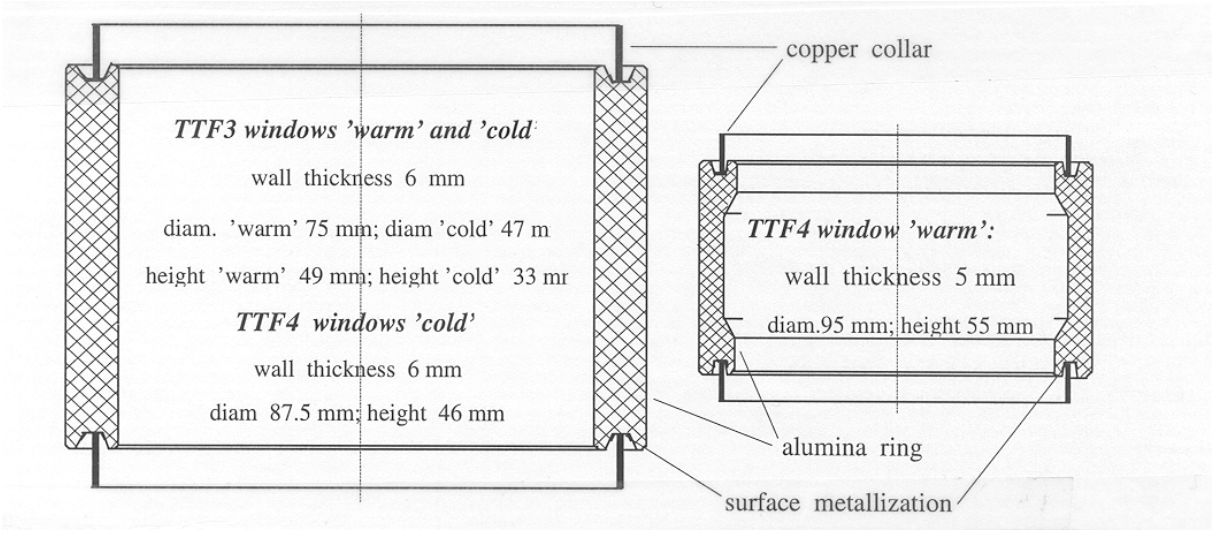
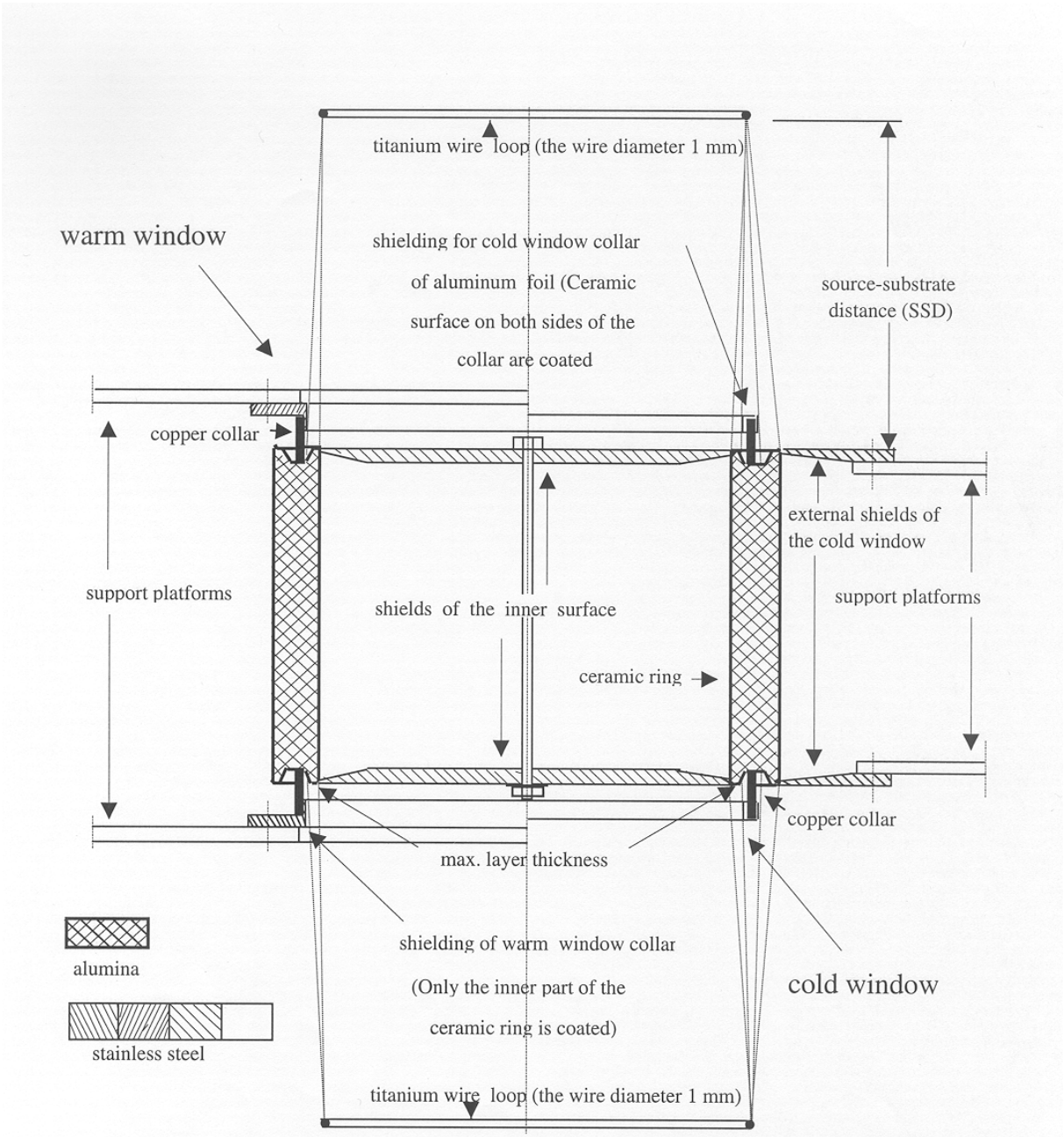



Figure 8: TTF3 and TTF4 cylindrical FR windows to be coated.

factor of ca 0.4) due to the shadow produced by the collar. Fortunately, the problem refers only to the cold windows and cannot be treated as layer non-homogeneity. The cold window surfaces on both sides of the collar are facing vacuum volumes , which are separated from each other (Figure 7). The layer thickness of 5 nm outside collar is thick enough to suppress any potential multipactoring whereas the corresponding 12 nm layer on the inner side cannot cause significant RF losses. The layer thickness inside the grooves (next to the metallization area) is further reduced due to low incidence angle at the groove walls. On the other hand, due to space restrictions and FR field distribution this area of the window‘s surface is not likely to become a place of multipactoring initiation. Computed titanium deposition rates for horizontal portions of the end surfaces at different filament currents are given in Figure 9.

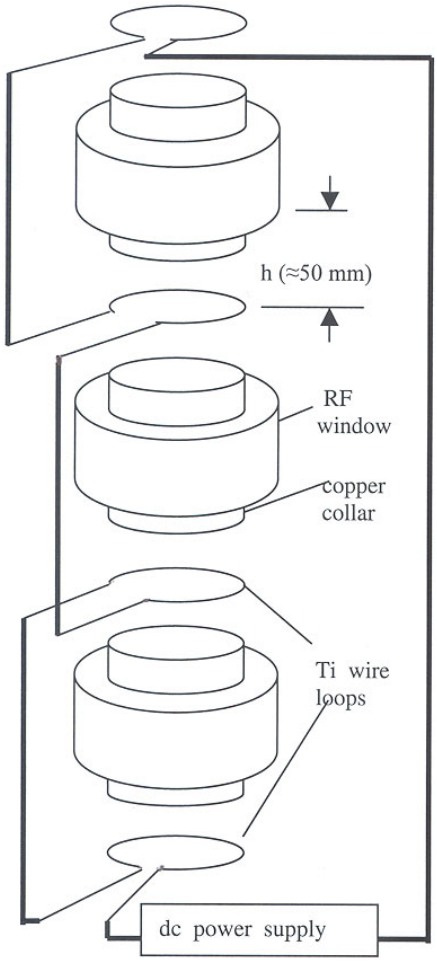
A sublimation setup for three, vertically arranged windows was used for surface processing of TTF4 coupler windows. The windows were coated from four titanium wire loops connected in series in a common circuit and powered from a single dc power supply. A general, simplified diagram is shown in Figure 10a. The smaller-size TTF3 windows (cold and warm alike) were in turn coated in a more integrated device consisting of three such circuits able to simultaneous surface processing of 9 pieces in a single operation, without breaking the vacuum. (Figure 10b). It consists of three platforms, 12 titanium wire loops connected in three separate circuits and a set of shields to protect the inner side of the ceramics from coating.



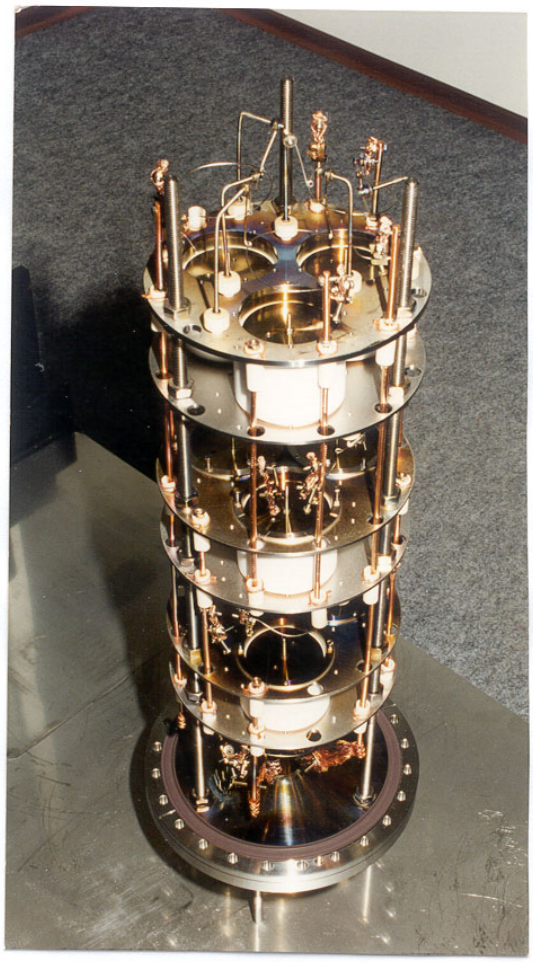

| Titanium deposition on end surfaces inside the collar (outside the collar for cold windows) | | | | | |
|---|---|---|---|---|---|
| source-substrate distance = 50 mm | | | | | |
| filament current (A) | 15 | 15.5 | 16 | 16.5 | 17 |
| deposition rate for cold TTF3 windows (nm/min) | 1.1 (0.4) | 1.7 (0.6) | 4.2 (1.5) | 8.3 (3) | 13.3 (4.8) |
| deposition rate for other windows (nm/min)* | 1.4 (0.5) | 2.2 (0.9) | 5.4 (2.1) | 11 (4.2) | 17 (6.7) |

* warm TTF3 windows, warm and cold TTF4 windows

Figure 9: Sublimation setup for end surfaces of warm (left side) and cold RF windows for TTF3 nad TTF4 couplers. The table contains calculated deposition rates.




(a)



(b)

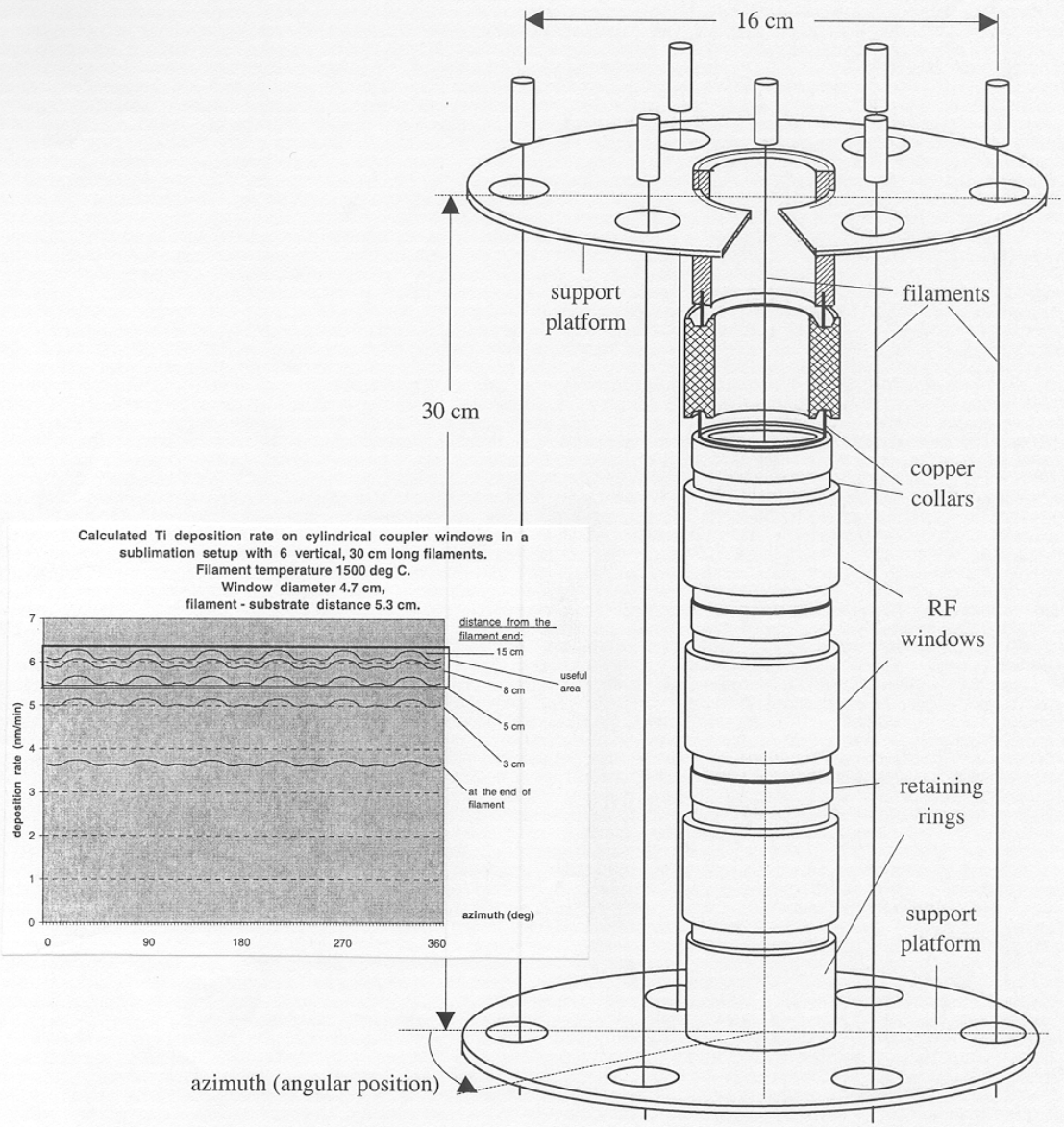
Figure 10: (a) Schematic diagram of the Ti sublimation setup for end surfaces coating showing the relative position of windows and filaments (the shielding and support structure are not shown) and (b) overall view of the integrated coating setup for TTF3 coupler windows.

**(2) Coating cylindrical surfaces**

Cylindrical surfaces were coated using a setup of 7 (9) vertical titanium wires (Figure 11) for TTF3 (TTF4) coupler windows. Up to five TTF3 windows could be coated, arranged axially in a vertical stake. One of the filaments was stretched along the axis of the windows to deliver titanium to their inner surfaces whereas the remaining six (eight for TTF4 windows) were arranged outside the stake as equidistant vertical lines, 8 cm off the stake axis. Numerical simulation of the layer distribution obtained in this setup revealed that for TTF3 cold windows the layer thickness do not vary over the external cylindrical surfaces by more than 10% of its average value (see the plot in Figure 11). In most cases the TiN layers obtained on these windows were optically transparent, uniform and grey-yellowish in color. To reach such layer uniformity the number and position of the external vertical filaments in this arrangement should be matched to the external diameter of the coated ceramic rings. In case of 6 filaments their distance from the window‘s axis should amount, as a rule of thumb, to 3-4 times the window radius. For a shorter source-substrate distance the number of filaments should be increased. Due to space restrictions in our vacuum vessel this condition could not be fulfilled for TTF4 cold windows with the ceramic ring radius bigger than 4 cm. In spite of increasing the number of outer filaments to 8 (their distance from the axis remained 8 cm) slight layer non-homogeneities were visible on some TTF4 cold windows after processing. To avoid the layer thickness drop near the filaments‘ ends, the filaments must extend above the top and below the bottom of the windows stake by a distance not shorter than the diameter of a single window.

A typical processing time schedule concerning ammonia admission, filament and substrate temperature is shown in Figure 4. Typical processing data for TTF3 and TTF4 windows for the setup shown in Figure 11 are given in Table 3. To avoid fast substrate cooling immediately after deposition and thus enhance Ti $\rightarrow$ TiN transition in the layer, the afterprocessing of TTF4 windows in ammonia was done in two steps: 30 min. at an ammonia pressure of 40 mbar and next – 0.5-3 days at 300-500 mbar.

Apart from complete TTF3 and TTF4 coupler windows, 4 ceramic rings (without copper collars) of TTF3 warm windows were surface-treated in the same way, the layer thickness ranging between 7 and 15 nm. The growth of their surface resistivities in air was monitored for over 10 months. The resistivity values reached GOhm region after 24 h
and 2 months for 7 and 15 nm-thick layers, respectively. Forty of the TiN – coated cylindrical windows for TTF3 and twenty for TTF4 [24] were installed in couplers in 2001 and in 2002, respectively. The details of TTF3 couplers processing (preconditioning) in RF field are given in [23]. The rest of the coated TTF3 windows are to be mounted in couplers by 2005.




| Titanium deposition on cylindrical surfaces of TTF3 and TTF4 windows using the above setup | | | | | |
|---|---|---|---|---|---|
| filament current (A) | 15 | 15.5 | 16 | 16.5 | 17 |
| TTF3 'cold', inner surface; deposition rate (nm/min) | 3.8 | 7 | 13 | 24 | 44 |
| TTF3 'cold', outer surface; deposition rate (nm/min)* | 1.8 | 3.3 | 6.2 | 11.5 | 21 |
| TTF3 'warm',inner surface;deposition rate (nm/min) | 2 | 3.7 | 6.8 | 12.7 | 23.3 |
| TTF4 'cold' and 'warm', inner surface; d.r. (nm/min) | 1.6 | 3 | 5.6 | 10.5 | 19.2 |
| TTF4 'cold', outer surface; deposition rate (nm/min)* | 2.3 | 4.2 | 7.8 | 14.5 | 26.6 |

* External surfaces of TTF3 and TTF4 windows were coated from 6 and 8 vertical filaments, respectively

Figure 11: Titanium deposition setup for cylindrical surfaces of TTF3 or TTF4 windows piled up in a vertical stack. The deposition rate distribution on TTF3 cold windows and average deposition rate values on different windows vs filament current are given in the plot and table.

Table 3: Recommended TiN coating parameters of TTF3 &4 cylindrical windows.

| Surface coated | Current (A) | Coating time (s) | Estimated max. layer thickness (nm) | Ammonia pressure (mbar) | | Processing time in $NH_3$ (days) |
|---|---|---|---|---|---|---|
| | | | | during deposition | after deposition | |
| TTF3 | | | | | | |
| end surfaces | 16 | 60 | 5 | $10^{-3}$ | 300 - 500 | 0.5 - 3 |
| inner | 16 (cold) | 30 (cold) | 6 (cold) | $10^{-3}$ | 300 - 500 | 0.5 - 3 |
| | 16 (warm) | 60 (warm) | 7 (warm) | $10^{-3}$ | 300 - 500 | 0.5 - 3 |
| outer | 16 (cold) | 70 (cold) | 7 (cold) | $10^{-3}$ | 300 - 500 | 0.5 - 3 |
| TTF4 | | | | | | |
| end surfaces | 16 | 50 | 5 | $10^{-3}$ | 300 - 500 | 0.5 - 3 |
| inner | 16 | 70 | 7 | $10^{-3}$ | 300 - 500 | 0.5 - 3 |
| outer | 16 | 50 | 6 | $10^{-3}$ | 300 - 500 | 0.5 - 3 |

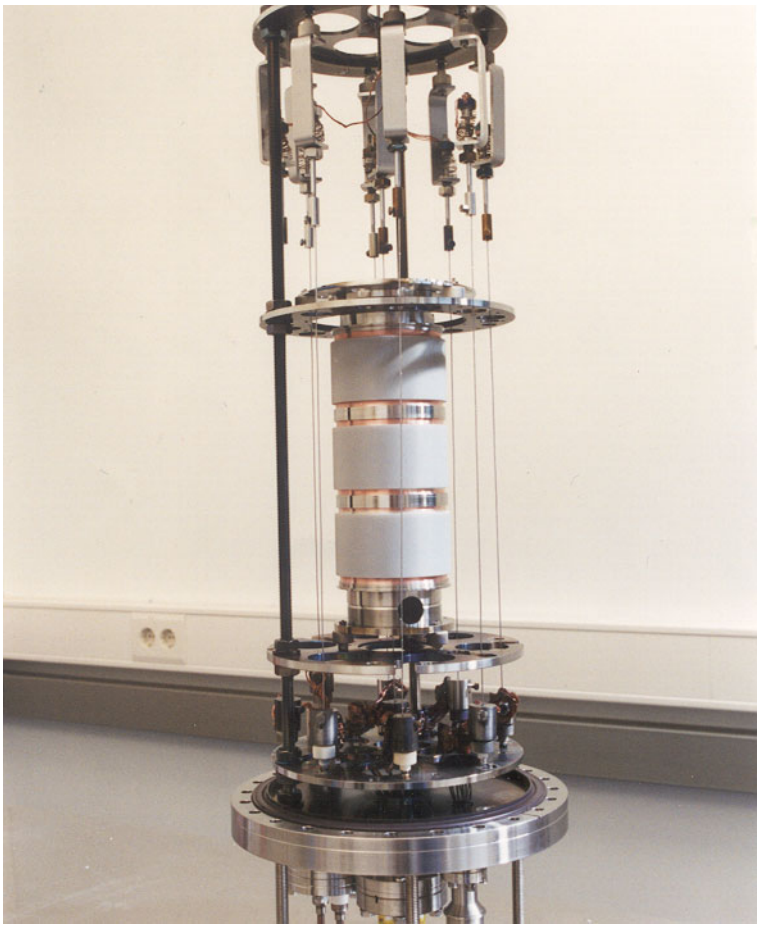


Figure 12: The arrangement for TiN coating of cylindrical surfaces of TTF4 windows.

## 4.2 TiN coating of TTF2 waveguide windows

The TTF2 coupler waveguide window consists of an aluminum disc (21.2 cm in diameter, 1 cm thick) installed in a stainless pillbox container (Figure 13). It works with atmospheric pressure on the one side and „warm“ vacuum on the other. The peak transmitted power during preconditioning is close to 1MW at a pulse length of 1.3 ms and repetition rate of several Hz. Lower than expected power transmission associated with light and electron emission on the vacuum side was due to multipactoring originated on aluminum disc or on metallic surfaces. With $TE_{10}$ waveguide transmission mode the multipactoring is likely to originate at the disc periphery. Therefore, sufficient TiN layer thickness should be reached at the rim of the disc and inside the pillbox, where the electrical RF field component normal to the disc is the highest. The layer thickness in the central part of the disc is not very crucial since the electric field is predominantly tangent to the ceramic surface in this region.

To obtain the required layer thickness distribution, deposition rate calculations have been performed for several possible filament shapes and positions. Due to space restrictions inside the pillbox reaching a uniform distribution of the deposit in a single operation (without breaking the vacuum) was impracticable. The distance between the disc and the opposite (parallel to the disc) pillbox wall is only 3 cm. In order to deliver a sufficient amount of titanium to the disc periphery the filament had to be installed inside the pillbox. Among possible shapes and positions of titanium filament the biggest titanium wire loop has been chosen (extreme dimensions of 16 cm x 14 cm) that could be introduced through WR650 waveguide opening (16.5 cm x 8.25 cm) in the pillbox wall. The distance between the loop and the disc was ca. 25 mm. The layer thickness computation showed that the required thickness distribution can be reached using this configuration (Figure 14). For a filament temperature of $1480^0$ C and deposition time equal 60 s the maximum calculated thickness of ca. 15 nm has been obtained on the disc in the region directly below the wire, which overlaps with the position of a large normal component of the RF electric field. The computed layer thickness at the disc periphery is close to 7 nm and the minimum thickness of below 5 nm is reached in its central region.

A suitable structure with current contacts and twelve electrically insulated filament supports has been manufactured (Figures 14-16). Preliminary deposition tests were performed in a test setup with precise simulation of the real pillbox shape, dimensions and the filament position. Due to a very short distance between the wire and the disc the layer thickness distribution changes rapidly with any vertical displacement of the wire caused by thermal stresses or high temperature creep. To avoid these effects the positions of the wire supports have been carefully adjusted (the distance between any neighbouring support points did not exceed 45 mm). Also the radius of curvature of the filament close to current contacts was optimized to avoid an excessively high layer thickness in this region. A direct thickness measurement in the test setup showed a pretty good agreement with the simulation results. The deposition rate varied from 10 to 15 nm/min in the regions close to the wire to ca. 3 nm/min in the center of the disc.

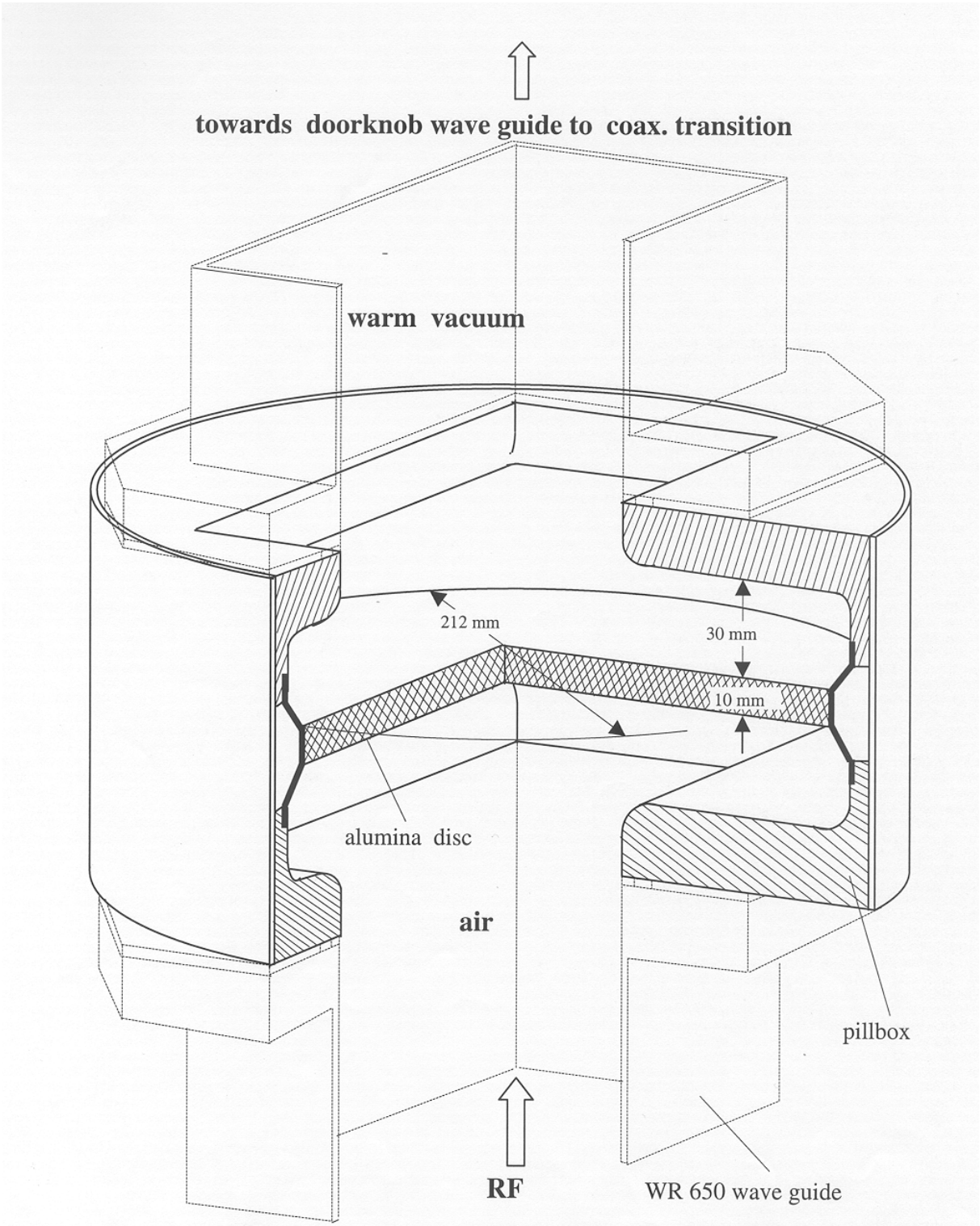
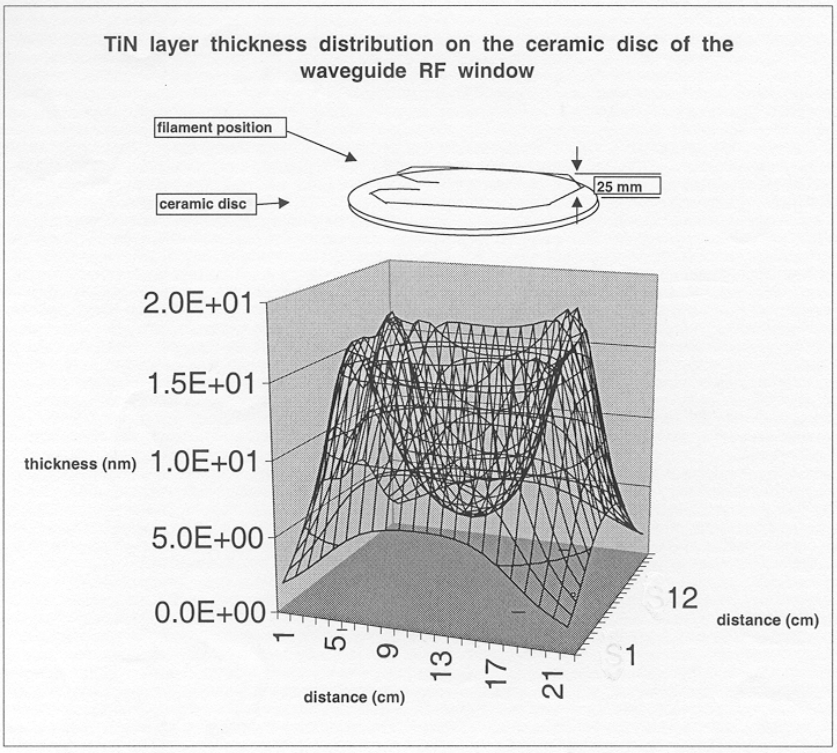
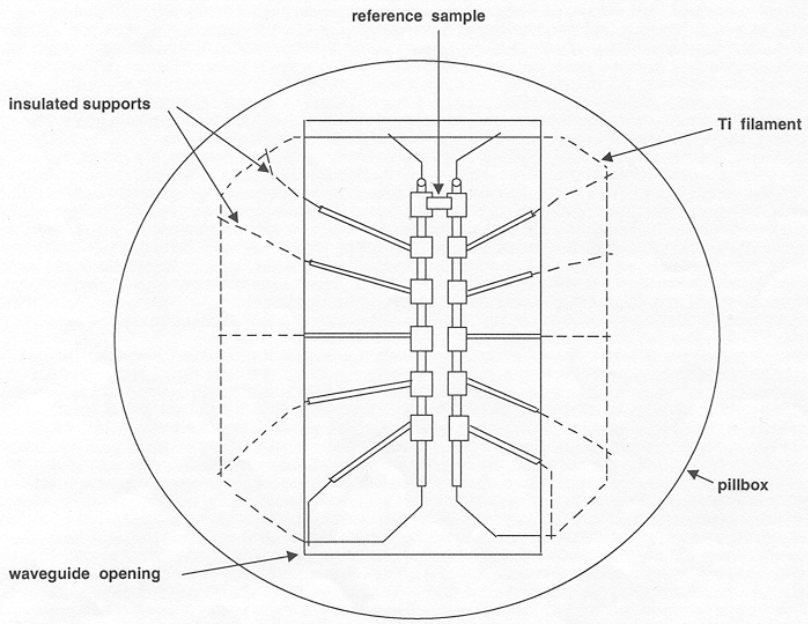



Figure 13: A cross-sectional view of the planar wave guide window of TTF2 coupler.




(a)




(b)

Figure 14: (a) TiN layer distribution on the ceramic disc of the TTF2 coupler wave guide window and (b) sublimation setup position in the pillbox.

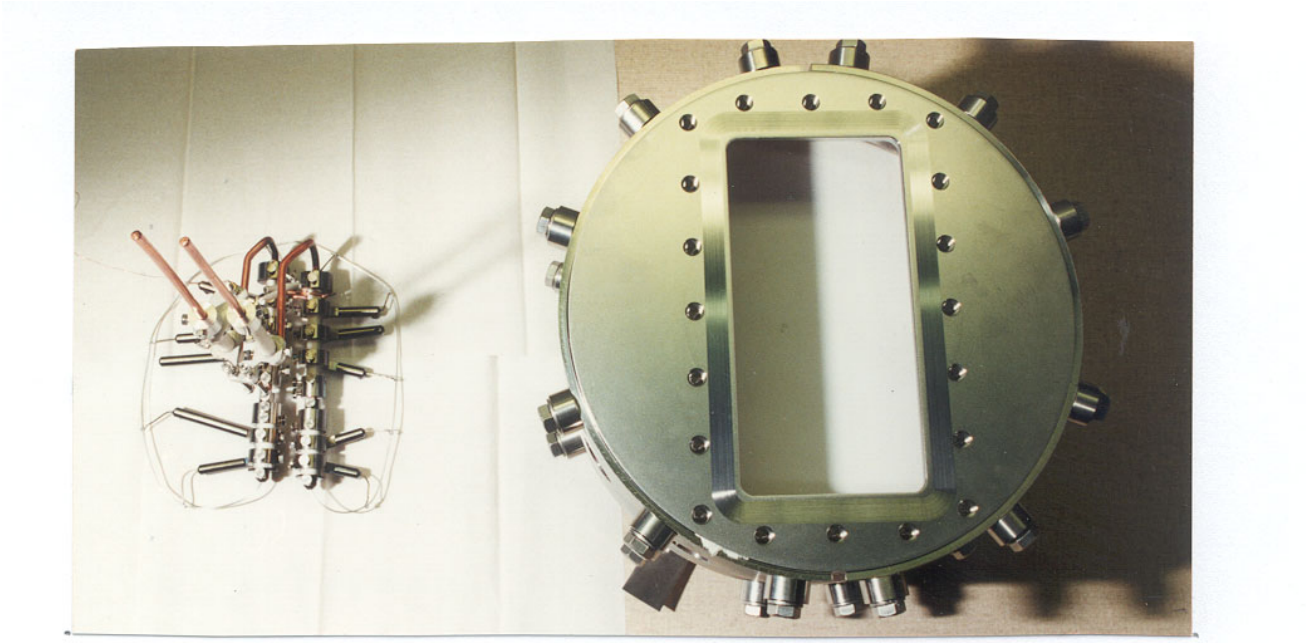


Figure 15: TTF2 coupler wave guide window (right) and the titanium sublimation assembly

Table 4: TiN coating parameters of TTF2 coupler wave-guide windows.

| Posi-tion | Number of windows coated | Filament current | Coating time | Layer thickness range | Ammonia pressure | | Colour of the disc surface |
|---|---|---|---|---|---|---|---|
| | | | | | during deposition | after deposition | |
| 1 | 2 | 17 A | 90 s | 7 – 30 nm | $2 \cdot 10^{-3}$ mbar | 950 mbar | metallic grey, pale in the center |
| 2 | 5 | 17 A | 60 s | 5 – 21 nm | $2 \cdot 10^{-3}$ mbar | 650 – 900 mbar | metallic grey, grey-yellowish, pale in the center |
| 3 | 14 | 16 A | 150 s | 5 – 20 nm | $2 \cdot 10^{-3}$ mbar | 300 – 700 mbar | grey yellowish, pale in the center |

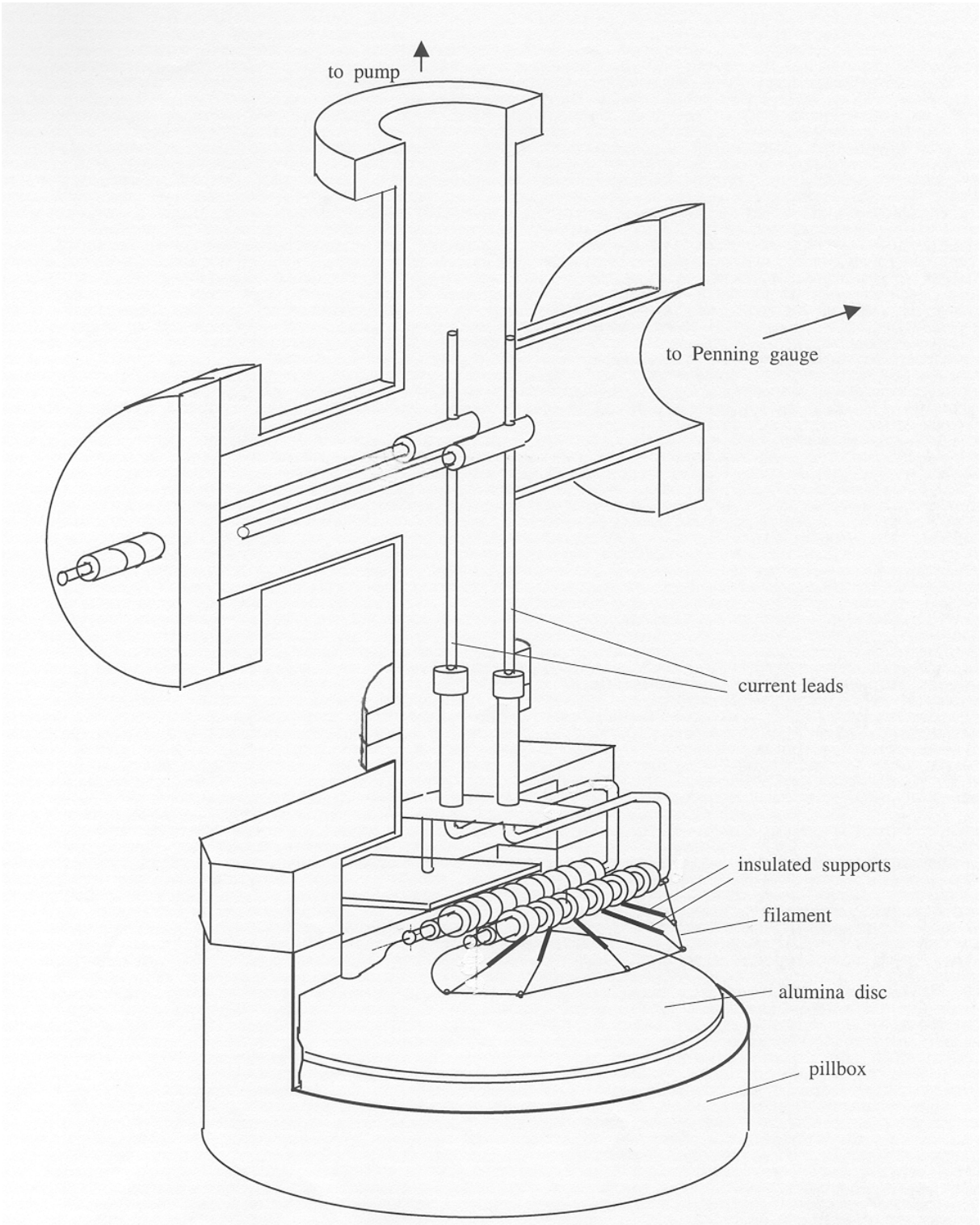



Figure 16: TiN coating assembly for TTF2 wave guide windows

21 wave-guide windows of TTF2 coupler have been TiN coated. Each time the filament assembly has been installed inside the pillbox using insulating supports. A reference ceramic sample was attached 22 mm off the filament to monitor the growth of layer resistivity (Figure 14 (b)). The window with the sublimation setup inside was equipped with a 60 mm diam. vacuum connection with an additional Penning gauge and attached to the pumping system (Figure 16). The surface treatment was done according to the timetable given in 2.3 except 30 min substrate preheating. To avoid thermal stresses filament current was rised in a stepwise manner before coating. The processing parameters are given in Table 4. The filament currents of 16 and 17 A corresponded to maximum deposition rates on the disc of 8 and 21 nm/min, respectively.

**RF performance of waveguide windows after surface processing**

The wave-guide windows performance after TiN coating improved significantly. Two windows (Table 4, pos. 1) were tested in a special RF test-stand at DESY and the others were installed directly in couplers. The impact of the coating on the windows' RF performance is summarized below:

- The necessary processing time has been reduced from a few to one day.
- No light or electron emission has been observed during RF operation after coating.
- RF performance became insensiive to a previous 1-day exposition to air.
- Proper power transmission has been reached.

No excessive power losses have been observed even in the case of locally high maximum layer thickness (30 nm; Table 4, pos.1) and its considerable residual conductance in the areas close to the filament. It may be explained taking into account the RF field distribution on the disc surface. The tangent component of electrical field is negligible in the area where the layer thickness attains maximum. Good results of the waveguide windows coating show that in such circumstances the lack of layer uniformity does not have to be a serious disadvantage assuming that the RF field distribution is taken into account in a proper way.

## 4.2 Whole surface TiN coating of TTF2 coupler

To further reduce secondary electron emission and thus the potential for multipactor, complete coating of a TTF2 coupler surfaces has been performed. Four main components of the coupler were surface treated separately (see Figure 7a):

- terminal waveguide section with doorknob waveguide to coaxial transition,
- inner and outer electrodes of warm coaxial 62 mm diam. line,
- complete 40 mm diam. cold coaxial line.

The stress was put on full surface coverage with titanium except hardly accessible areas (like bellows ect). As in the case of ceramic components the Ti layer was converted during deposition to TiN under ammonia flow at a pressure of $(1\text{-}2)10^{-3}$ mbar. In the case of the TTF2 coupler coating the estimated thickness varied between 10 and 25 nm [25]. For further Ti→TiN conversion the titanium coated components were treated in ammonia of 300-600 mbar presure after deposition. Adequate sublimation setups and processing parameters are listed in Table 5 and shown in Figures 17-18.The sublimation setup for cold coax. was composed of eight vertical filaments supported by four insulated titanium rods inserted between inner and outer electrodes (Figure 18b). Full coverage with titanium of the surfaces was reached. Flexibility of supporting rods and an incidental short in one of the filaments resulted in visible lack of homogeneity in some locations on the antena surface. A more rigid support structure for filaments must be used in the future for titanium deposition in this critical area.

Table 5: TiN coating arrangements of TTF2 coupler components.

| TTF2 component | Sublimation setup | Figure no. | Processing parameters | | |
|---|---|---|---|---|---|
| | | | current | coating time | estimated layer thickness |
| doorknob waveguide to coax. transition | a single titanium wire loop inside the waveguide | 17 | 17 A | 80 s | 10 - 20 nm |
| external conductor of the warm coax | a single filament installed axially | | 17 A | 70 s | 10 - 20 nm |
| internal conductor of the warm coax | six vertical 35 cm-long filaments, 8 cm from the axis | 18a | 17 A | 70 s | 8 - 16 nm |
| complete cold coax | eight vertical filaments installed inside the line half-way between the external conductor and the antenna | 18b | 14 A | 150 s | 6-25 nm |

RF check of the coated coupler showed a very short processing time (4.5 hours) needed to reach 1MW stable power regime at a pulse length of 20μs and repetition rate 2 Hz. Reaching the standard maximum tested power level (1MW at a pulse length 1.3 ms) took extra 24 h [23]. After extending the power load up to 1.8 MW, however, electron emission

started in a strictly localized area of the antenna surface, at a pulse length of 0.8 ms. It could not be suppressed by further RF processing. The antenna surface was partially destroyed and metallic deposits (ca 1 atomic % of Cu and Mg) were found on the rim of the cold window.

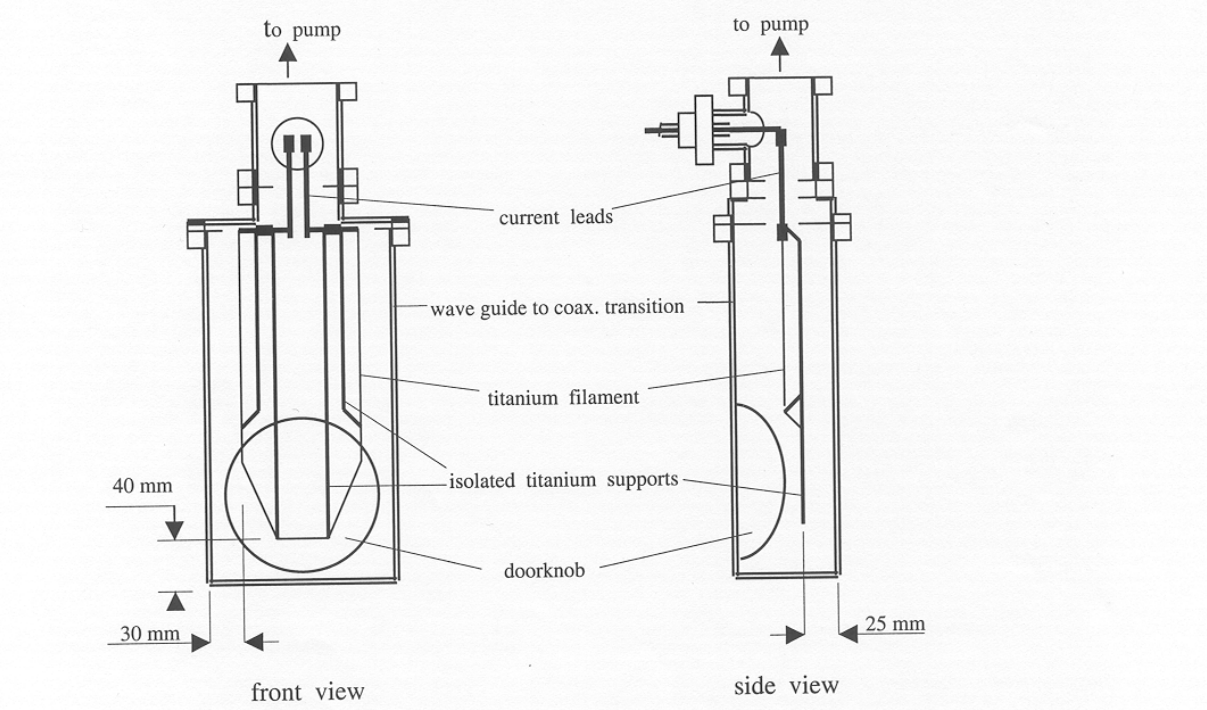



Figure 17: Schematic view of the sublimation setup for TiN coating of the wave guide to coax. transition of TTF2 coupler.

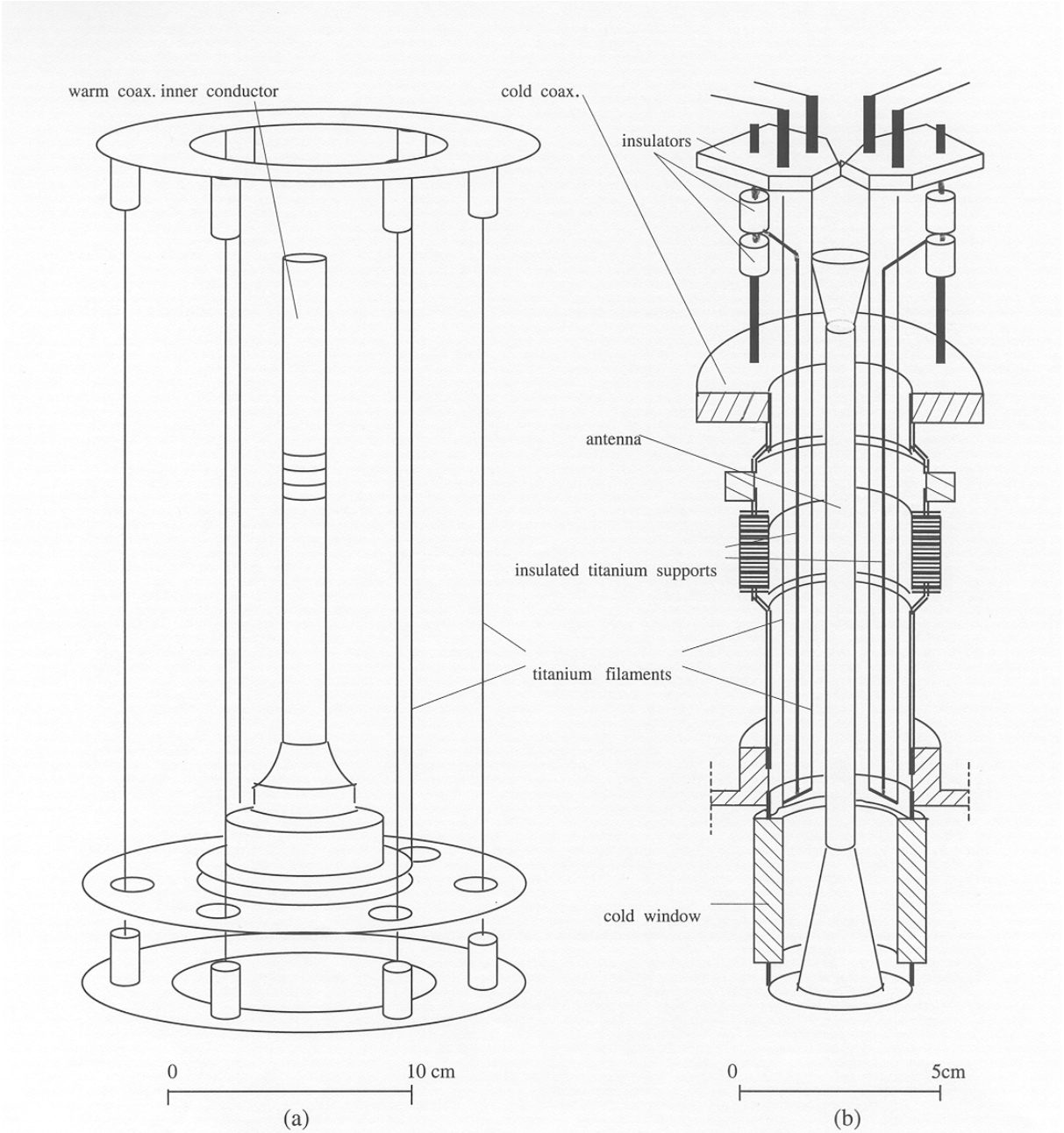



Figure 18: Tianium coating arrangements for (a) warm coax. inner conductor and (b) cold coax. of TTF2 coupler. The upper contacts of the filaments are supplied with spring mechanisms to stabilize the wire position.

# 5 Concluding remarks

The sublimation TiN-coating technique has been used for anti – multipactor protection of TESLA couplers. A significant improvement was achieved as to RF performance of TTF2 waveguide windows. Also the complete TiN coverage of the TTF2 coupler resulted in a strong reduction of the conditioning time necessary to reach a stable operation at 1 MW maximum power and 1.3 ms pulse length in a test-stand at room temperature. Nevertheless, to avoid coupler destruction strict power limitation must be observed.

Sublimation steups have been designed and tested for processing of various types of RF coupler windows as well as other standard coupler components (elements of coaxial lines, waveguide sections ect). The processing parameters were optimized. They can be used on a larger scale in the future for anti-multipactor surface coating for Tesla Test Facility. Extension of the TiN-coating activity requires, however,using a new, larger vacuum chamber that would enable reaching uniform deposition rate over large areas, using more diagnostic tools (eg crystal quartz thickness monitor) and facilitating the setup assembly.

## Acknowledgments

Thanks are due to all the many colleagues involved in preparation of the coating setup and RF preconditioning tests of the couplers with TiN-coated components. The valuable collaboration of Mrs. Katrin Lando (DESY) in manuscript preparation is acknowledged.

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# Supplement: TTF3 coupler windows coating manual

Deatiled description of the TiN coating procedure of TTF3 RF windows is given for both the end surfaces (pp. 35-37) and the cylindrical surfaces (pp. 38-40) coating. The mounting of the sublimation setup is normally preceeded by cleaning of the device in ultrasonic bath in a degreasing agent (eg „Ticopur") and next in one or more baths – in ultra-pure water, followed by drying in pure nitrogen blow.

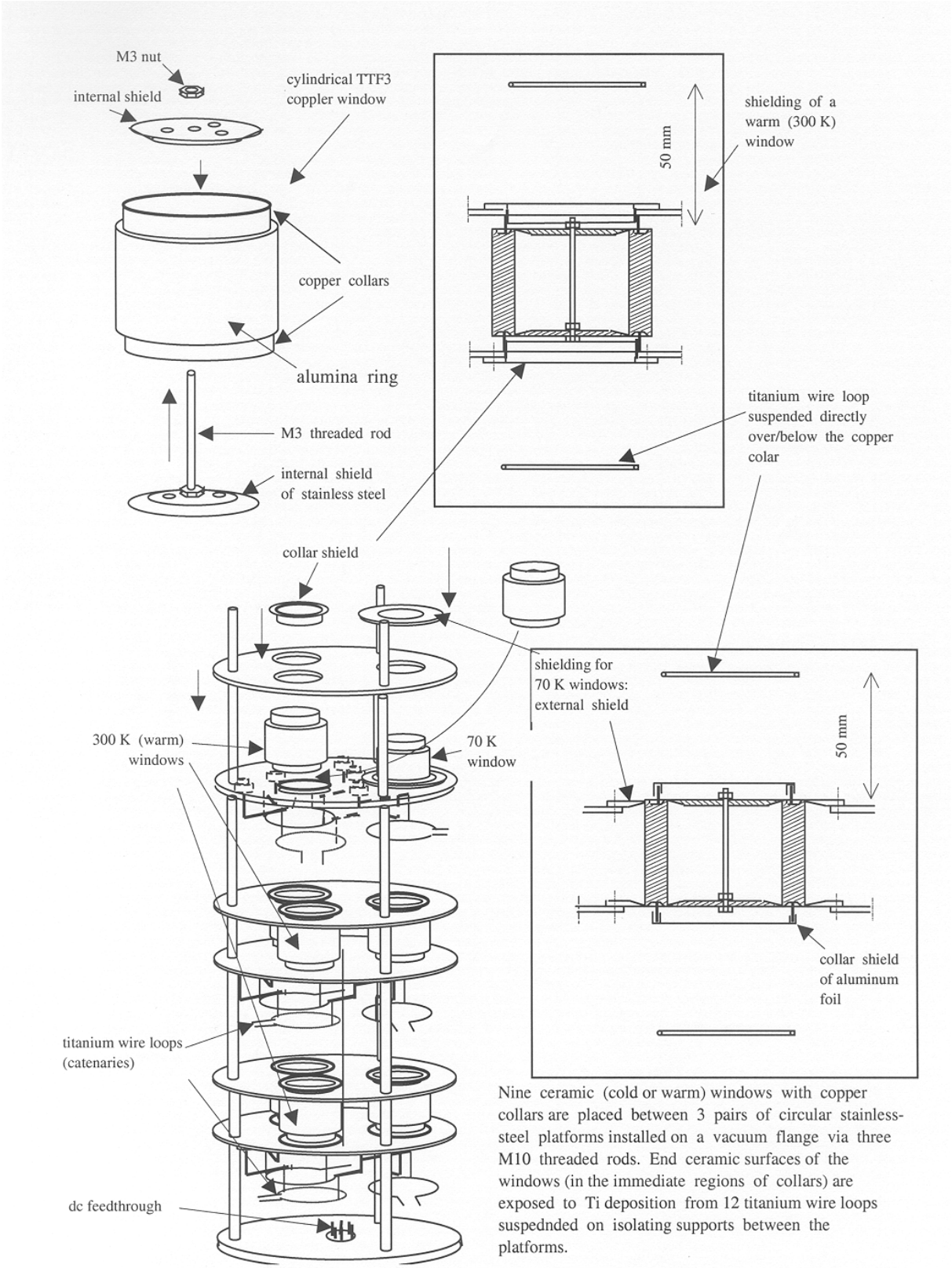


Fig. 19: The first assembly step of a sublimation setup for TiN coating of end window surfaces. Simultaneous coating of cold (70 K) and warm windows is not possible.

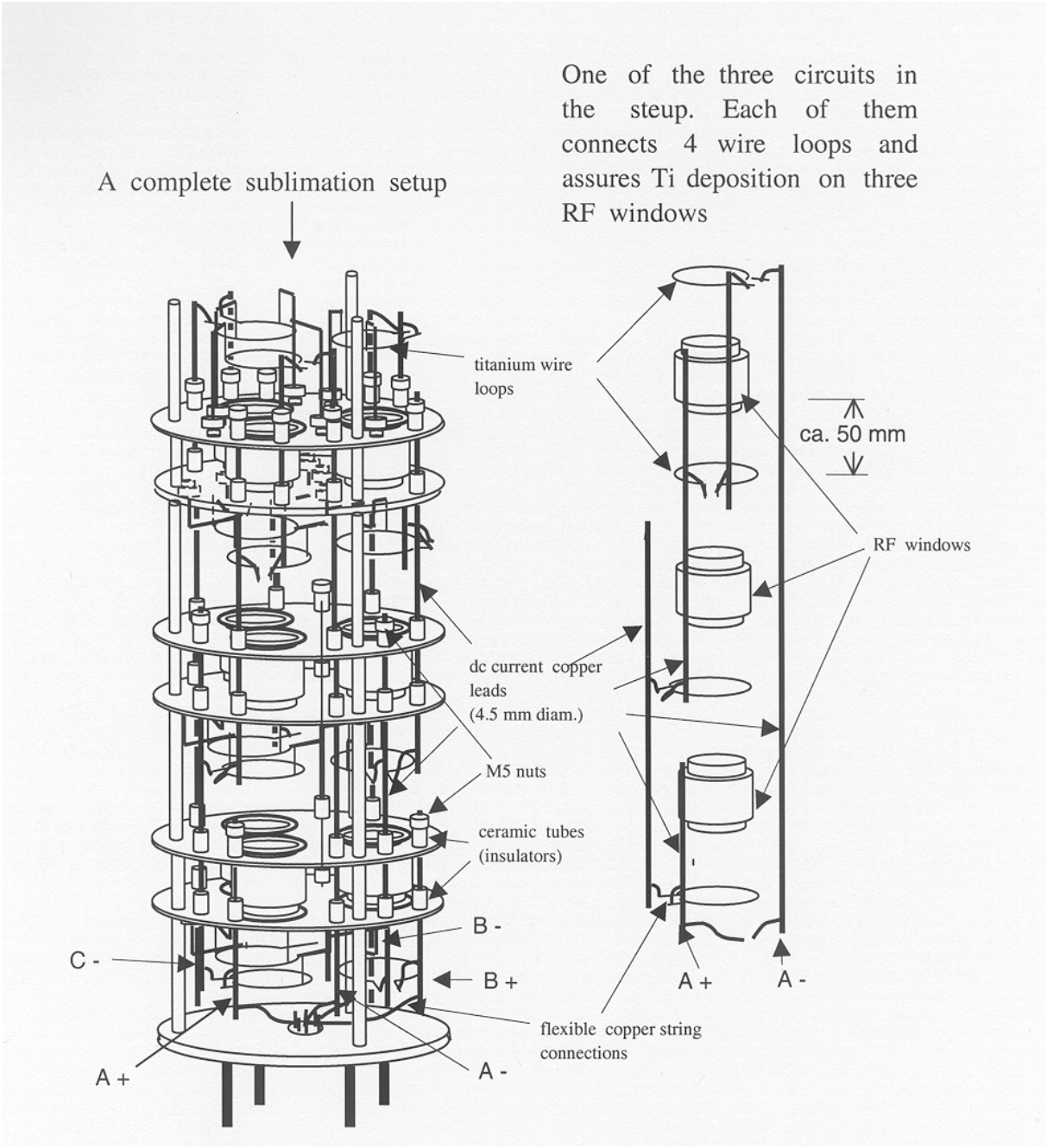


Figure 20: dc current connections in the sublimation setup for the end surfaces coating.

The dc installation of the setup consists of three separate circuits, each of which connects 4 titanium wire loops capable of simultaneous coating of the end surfaces of 3 windows. The circuits are further referred to as circuits „A", „B", and „C".

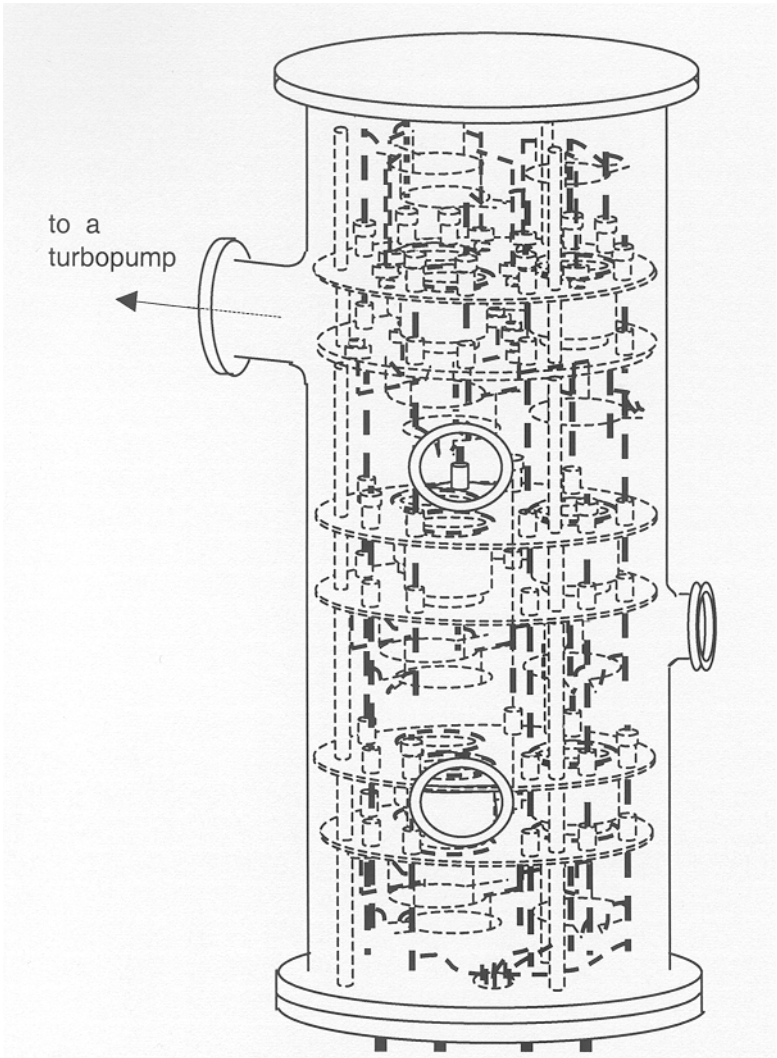


Figure 21: Sublimation setup installed inside the vacuum chamber connected to the turbopump unit.

## TiN coating of the end surfaces of cylindrical coupler windows – time schedule:

1. The vacuum vessel is pumped down to a pressure of $(2 – 5)\ 10^{-6}$ mbar.
2. The vessel is filled with ammonia (99.8% purity) up to a pressure of $10^{-3}$ mbar and pumped out again.
3. All the titanium wire loops are heated with a dc current of ca. 8 A for 30 minutes.
4. A steady ammonia flow is established under continuous pumping at a pressure of $10^{-3}$ mbar; „A", „B" and „C" circuits are fed one-by-one with dc current of 16 A for 60 s each.
5. A cut-off valve between the pumping unit and the vessel is closed, the vessel is filled with ammonia up to 10 mbar and kept for 30 min.
6. The ammonia pressure is then rised up to 200 mbar and kept for at least 6 hours.
7. The ammonia is pumped out through a by-pass pumping connection using a forevacuum pump, the vessel is filled with pure nitrogen and then exposed to air. The windows are extracted from the setup.

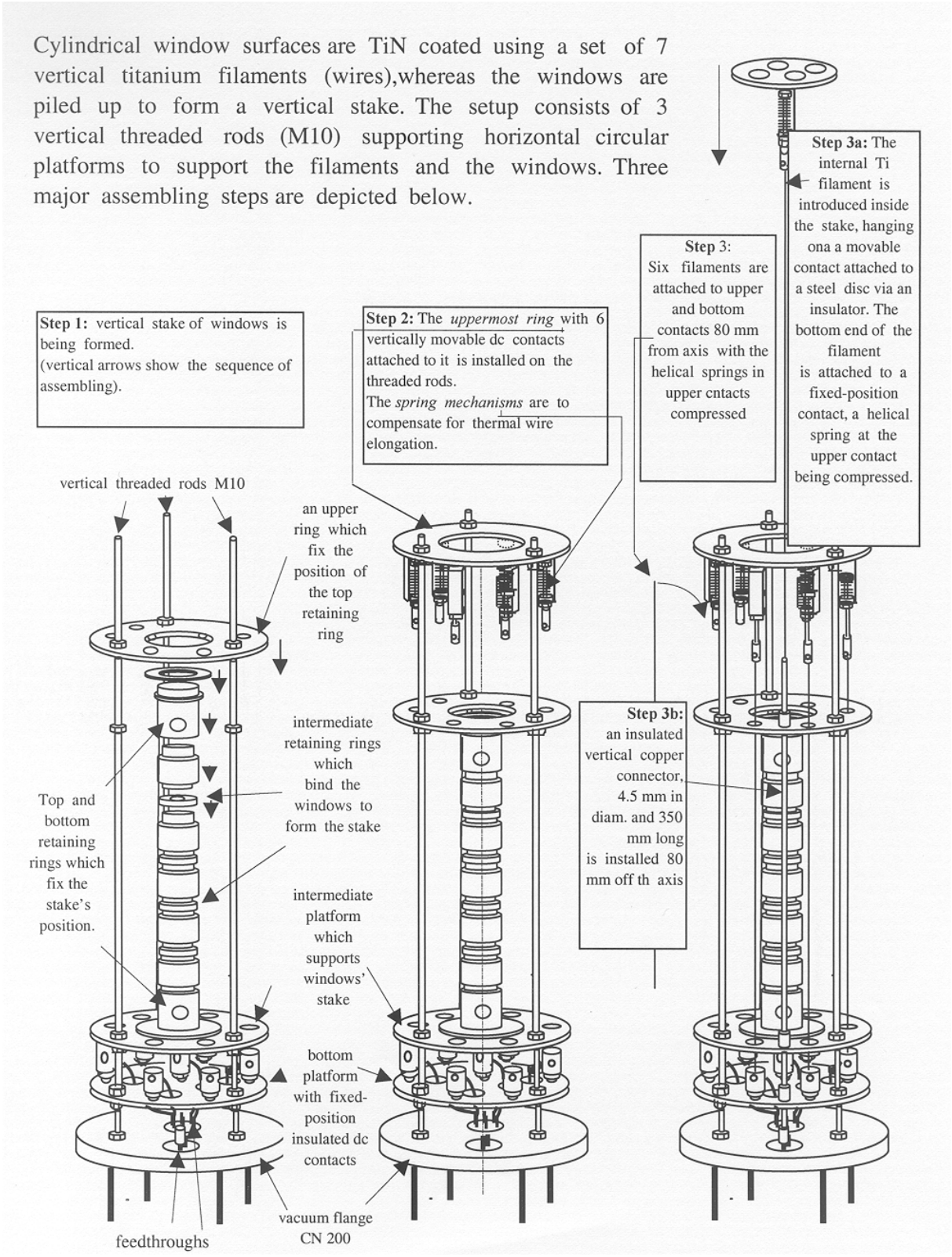


Figure22: Three steps of assembling the sublimation setup for cylindrical surfaces coating.

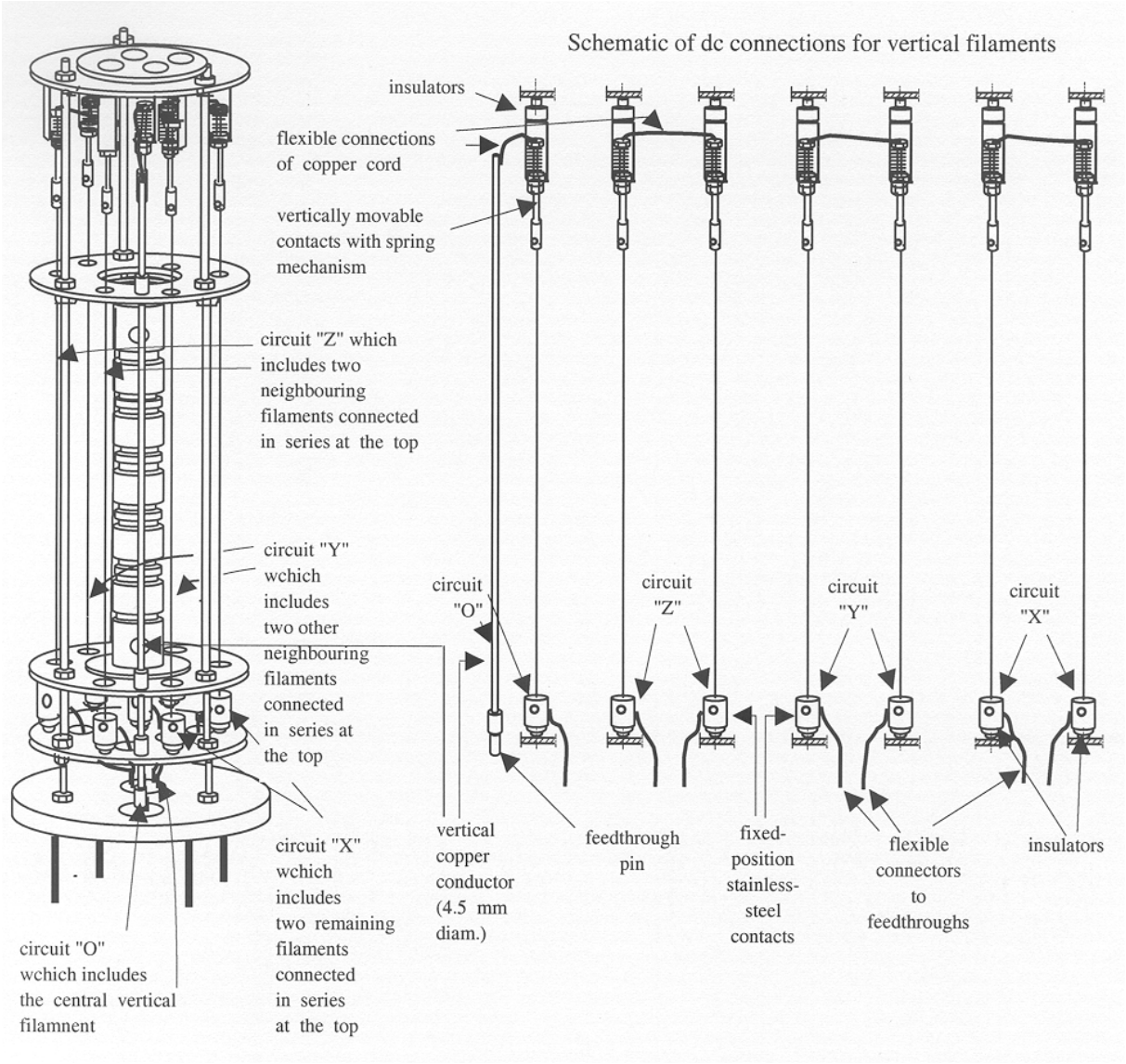


Figure 23: Complete sublimation setup for cylindrical surfaces coating and its dc connection schematic.

The sublimation setup for the final operation of cylindrical surfaces TiN coating is done according to the above schemes. Before introducing into the vacuum chamber the setup is checked against possible shorts and disconnections. The internal filament is fed via a copper rod, 4.5 mm in diam., connected to a pin of an additional dc feedthrough installed 80 cm off the axis. The top of the rod and the movable contact of the inner wire are linked by a flexible connection of copper string, 3mm in diam. The circuit used for feeding the central wire is further referred to as the circuit „0". The top movable contacts of the remaining filaments (installed 80 mm off the axis) are connected in pairs by copper strings. The three dc current circuits used for heating these filamnets are called circuit „X", circuit „Y" and circuit „Z". The corresponding feedthrough pins are marked by X+, X-, Y+, Y-, Z+ and Z-, correspondingly.

**All the instalations are performed using vacuum gloves!**

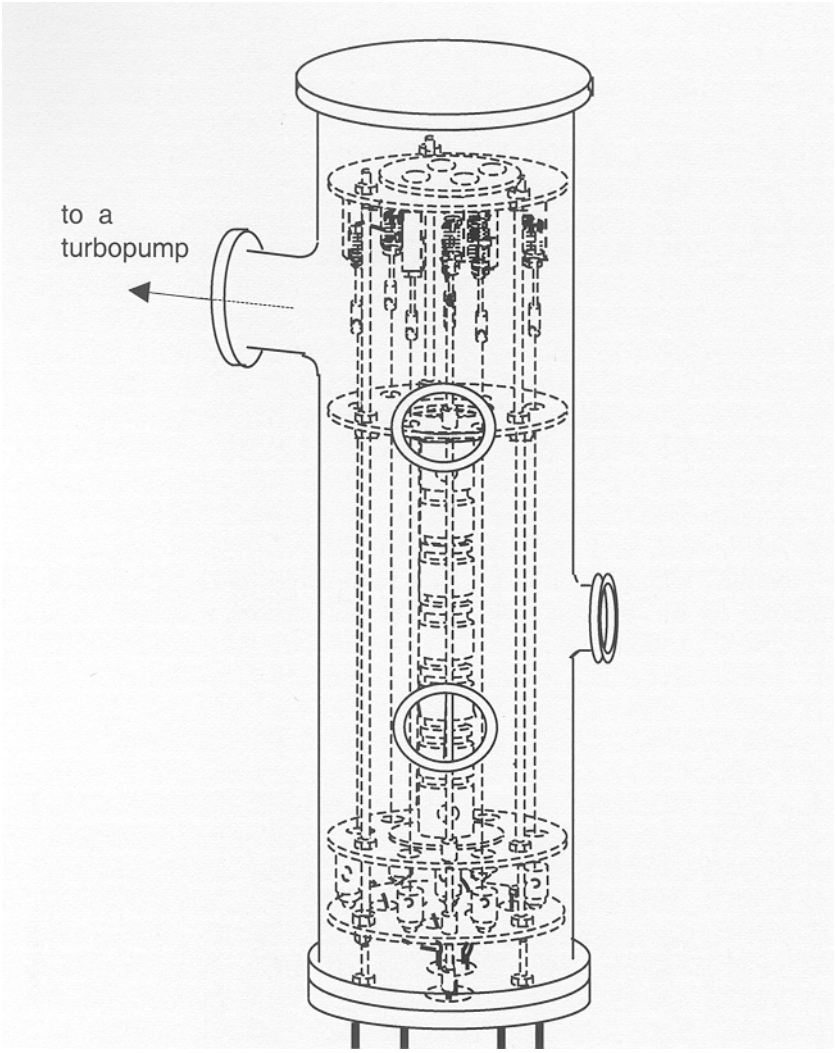


Figure 24: Sublimation setup for TiN coating of cylindrical surfaces in the vacuum.

## Coating time schedule:

1. The vessel is pumped down to ca $5 \cdot 10^{-6}$ mbar, then – vented with $NH_3$ (to $10^{-3}$ mbar) and pumped again to reach the basic vacuum.
2. The filaments are heated with dc current of ca. 8 A for 30 min.
3. The vessel is filled with ammonia up to $10^{-3}$ mbar under continuous pumping.
4. For warm (300 K) windows coating the circuit „0“ is biased with a voltage of +10 V respectively the ground, the current in the circuit „0“ is rised up to 16 A, maintained for 45-50 s and then switched off. In the case of cold windows the circuit „0“ is fed with a dc current of 16 A for 30 s (after applying the positive bias as above) and next switched off. Then each of the „X“, „Y“ and „Z“ circuits are one-by –one biased with +10 V and fed with a current of 16 A for 50 s.
5. After closing the cut-off valve leading to the pump the ammonia pressure is rised to 10 mbar. After 30 min. it is rised further to 200 mbar
6. After 12 to 72 hours the ammonia is pumped out by the forevacuum pump through the by-pass connection, filled with pure nitrogen and exposed to air.